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TITLE: One component room temperature curable sealant compositionAbstract Text (1):A one component room temperature curable sealant composition comprising:Brief Summary Text (3):

The present invention relates to a one component room temperature curable sealant composition. Particularly, it relates to a one component room temperature curable sealant composition which is useful as a sealing material, an adhesive or an injection molding material and which exhibits good adhesion to various substrates including metal, glass and coated steel sheets and plastics such as a vinyl chloride resin, an acrylic resin or a styrol resin, without requiring a primer coating. More particularly, the composition of the present invention is widely useful as a sealant for various applications, for instance, for sealing the joint of interior or exterior building structures, repairing cracks in mortar or concrete structures, fixing the windshields of automobiles, sealing seams of joined steel sheets, etc., and it can advantageously be applied directly to substrate surfaces such as metal, glass, melamine and acrylic resin surfaces without employing a primer.

Brief Summary Text (5):

As a one component room temperature curable sealant, a silicone or polyurethane sealant is well known. The silicone sealant has advantages such that it has good weather resistance and heat resistance, and its curing speed is high and yet its storage stability is good. On the other hand, it has disadvantages such that a coating composition is hardly applied onto a cured silicone sealant, and it is likely to stain stone materials. Whereas the polyurethane sealant has advantages such that it is superior to the silicone sealant in the curing properties, and it has good comparability with a coating composition and its production costs are relatively low. On the other hand, the polyurethane sealant has drawbacks such that it is inferior in the weather resistance and heat resistance, and its storage stability tends to be poor when it is made to have a high curing speed.

Brief Summary Text (7):

Heretofore, with respect to a one component room temperature curable composition for a sealing material, a coating material, an adhesive and an injection molding material, there has been proposed a composition comprising (A) an addition product of a polyether urethane prepolymer having terminal isocyanate groups and .gamma.-amino-propyltrimethoxysilane and (B) a small amount of N-.beta.(aminoethyl)-.gamma.-amino propyltrimethoxysilane (Japanese Examined Patent Publication No. 5061/1978) as the material containing as the basic component a polyether polymer having a hydrolyzable silicone functional group as its terminal group.

Brief Summary Text (8):

Further, there has been proposed a composition containing as the main component an oxypropylene polymer having a terminal silylether group represented by the formula: ##STR5## (Japanese Unexamined Patent Publications No. 156599/1975 and No. 73998/1977).

Brief Summary Text (9):

However, these compositions had a disadvantage such that they are hardly bonded to the above-mentioned substrates without a primer coating.

Brief Summary Text (10):

of the polymerization, during the polymerization or to the starting materials. Usually, the mixture of the polymer and the compound (V.II) is dispersed in water by being stirred in. Such dispersions are generally known and are described for example in EP-A-467 908.

Suitable compounds (V.II) are araliphatic C_{60} - C_{30} diamines or aliphatic C_{60} - C_{30} polyamines, preferably diamines or triamines, and polyesters of a C_{20} -alkyl alcohol and a C_{20} -thiomonocarboxylic acid, eg. pen-taerythrityl tetrakis-3-mercaptopropionate.

The novel polyurethane dispersion may contain further water-emulsifiable or water-dispersible resins, such as poly-mer resins, polyurethanes, polyester resins, epoxy resins or alkylid resins, and commercial assistants and additives, such as blowing agents, emulsifiers, thickeners and

They are suitable, for example, for adhesive bonding or coating of different substrates, such as wood, metal, plastics, paper, leather or textile, and for the production of moldings and printing inks.

The novel polyurethane dispersions can be processed by the methods usually used in the adhesives and coating industries, ie. by applying the dispersions to the substrate by spraying, rolling or knife coating and then carrying out drying.

For processing as an adhesive, the coated workpieces are preferably joined to another workpiece with application of pressure, either before drying of the dispersion film or after drying.

Particularly strong adhesive bonds are obtained if work-pieces which are provided with a dried adhesive film are heated to about 50°-100° C. immediately before, during or after joining.

The adhesive bonds produced by these methods are distinguished in particular by the fact that they are stable and make it possible to produce bonds having high heat distortion resistance.

EXAMPLES

Preparation of a polyurethane dispersion containing a disperse phase P.Ia

Example 1

530 g of a polypropylene oxide diol having an OH number of 56 were reacted with 0.5 g of DBTL, 21.4 g of DMPA, 27.5 g of neopentylglycol and 140.8 g of TDI at 110° C. for 2 hours. After dilution with 880 g of acetone and cooling to 50° C., the NCO value was 0.6%. After neutralization with 17.9 g of 25% strength NaOH, 380 g of a 50% strength acetone solution of an adduct of 2 mol of acrylic acid with 1 mol of bisphenol A bisglycidyl ether were added and the mixture was dispersed with 1200 g of demineralized water. The acetone was distilled off under reduced pressure.

Acrylate content: 0.86 mol/kg solid

Analytical data:

FC: 40%

LD: 95

pH: 8.2

Viscosity: 373 mPas

K value in NMP: 44

Example 2

565.5 g of a polyester of adipic acid and butanediol, having an OH number of 45 were reacted with 0.15 g of DBTL and 29. g of TDI in 152 g of acetone for 1 hour at 65° C. Thereafter, 28.6 g of HDI were metered in and the mixture was kept at this temperature for a further 37 minutes. Dilution was effected with 609.9 g of acetone and the mixture was cooled to 50° C. The NCO value was 0.65%. Chain extension was carried out with 40.9 g of PUD salt, and 380 g of a 50% strength acetone solution of an adduct of 2 mol of acrylic acid with one mol of bisphenol A bisglycidyl ether were added. Dispersing was then effected with 1200 g of demineralized water. After the acetone had been distilled off under reduced pressure, the mixture was brought to a solids content of 40%.

Acrylate content: 0.826 mol/kg

Analytical data:

FC: 40%

LD: 52

Viscosity: 26 mPas

pH: 8.8

K value: 69

Example 3

605.2 g of polypropylene oxide diol (OHN=56), 0.5 g of DBTL, 21.5 g of DMPA and 14.8 g of TDI for 2 hours at 110° C. 100 g of acetone, 0.1 g of hydroquinone dimethyl ether and 18.6 g of hydroxyethyl acrylate were added and the reaction was continued for a further 2 hours at 90° C. After dilution with 878 g of acetone and cooling to 50° C., the NCO content was 0.48%. Neutralization was effected with 6.4 g of NaOH dissolved in 20 g of demineralized water and dispersing was carried out with 1200 g of demineralized water. The acetone was distilled off under reduced pressure.

Acrylate content: 0.2 mol/kg solid

Analytical data:

FC: 40%

LD: 91

Viscosity: 380 mPas

pH: 8.7

K value: 33

Example 4

385.3 g of a polyesterol obtained from adipic acid and ethylene glycol (OHN=42), 0.5 g of DBTL, 21.45 g of DMPA, 0.1 g of dimethylhydroquinone and 269.7 g of an adduct of 2 mol of acrylic acid with 1 mol of 1,4-butanediol bisglycidyl ether (Mn=330) were initially taken and reacted with 123.5 g of TDI for 1 hour at 110° C. Dilution was effected with 977 g of acetone and the mixture was cooled to 50° C. The NCO value had decreased to 0. Neutralization was effected with 6.4 g of NaOH in 20 g of demineralized water and dispersing was carried out with 1200 g of demineralized water. The acetone was distilled off under reduced pressure and the solids content was brought to 40%.

Acrylate content: 2 mol/kg solid

Analytical data:

FC: 40%

LD: 94

Viscosity: 38 mPas

pH: 7.9

K value: 32

Preparation of a polyurethane dispersion containing a disperse phase P.II

Example 5

595.1 g of polypropylene oxide diol having an OH number of 56, 0.5 g of DBTL, 30.9 g of neopentylglycol and

As additives to improve the adhesion of the polyether polymer having at its terminal a hydrolyzable silicone functional group, there have been known a phenol resin, an epoxy resin (Japanese Unexamined Patent Publications No. 73729/1977 and No. 156599/1975), a polyisocyanate compound having an active isocyanate group (Japanese Unexamined Patent Publication No. 73729/1980) and a triazine ring compound (Japanese Unexamined Patent Publication No. 34154/1982). However, these additives do not necessarily provide adequate effectiveness and have drawbacks such that they are inferior in the water-resistant bonding and durability, whereby there have been problems such that stabilized bond strength is hardly obtainable due to restrictions with respect to the curing catalyst or the mixing conditions, and that no adequate storage stability is obtainable as a one component sealant.

Brief Summary Text (13):

The present inventors have conducted extensive researches to overcome the above-mentioned drawbacks of the conventional sealants and to provide a one component room temperature curable sealant composition which can be applied to various substrates without requiring a primer and which, at the same time, has good water-resistant bond strength, durability and storage stability. As a result, the present invention has been accomplished.

Brief Summary Text (14):

Namely, the present invention provides a one component room temperature curable sealant composition comprising the following components (A), (B) and (C):

Brief Summary Text (33):

The organic titanic acid ester (e) in the present invention serves to substantially improve the bonding when used in combination with the aminoalkoxysilane (a). As specific examples of the organic titanic acid ester, there may be mentioned tetraisopropyl titanate, tetra-n-butyl titanate, butyl titanate dimer and tetra (2-ethylhexyl) titanate. The organic titanic acid ester (e) is added in an amount of from 0.1 to 1.2 mols, preferably from 0.3 to 1.0 mol, relative to the aminoalkylalkoxysilane (a). Stabilized bonding will be hardly obtainable if the amount of the organic titanic acid ester is less or more than the above mentioned range.

Brief Summary Text (35):

The amount of the partial addition condensation product obtained by the reaction of these materials, is preferably within a range of from 0.5 to 10 parts by weight relative to 100 parts by weight of the polyether polymer (A) having a hydrolyzable silicone functional terminal group. If the amount is less than 0.5 part by weight, no adequate effectiveness of the addition is obtainable. On the other hand, if the amount exceeds 10 parts by weight, the curing properties of the sealant composition tend to be impaired and the cost will be increased.

Brief Summary Text (38):

Further, an oxide of a metal belonging to Group II of Periodic Table such as zinc oxide, calcium oxide or magnesium oxide and/or an organic primary amine such as xylylenediamine, hexamethylenediamine or octylamine may be incorporated to the sealant composition of the present invention, as a co-catalyst to adjust the curing speed, as the case requires.

Brief Summary Text (39):

Furthermore, a filler, a plasticizer, a pigment, an aging-preventive agent, a ultra violet ray absorbing agent or a viscosity controlling agent may also be added to the composition of the present invention depending upon the particular purpose of the composition.

Brief Summary Text (41):

As the plasticizer, there may be used commonly employed plasticizers, for instance, phthalic acid esters such as dioctylphthalate, dibutylphthalate or butyl benzyl phthalate; aliphatic dibasic acid esters such as dioctyl adipate, isodecyl succinate or dibutyl sebacate; glycol esters such as ethyleneglycol dibenzoate or pentaerythritol ester; aliphatic esters such as butyl oleate or methyl acetylricinolate; phosphoric acid esters such as tricresyl phosphate or trioctyl phosphate; and chlorinated paraffins. Further, a liquid resin having a molecular weight of from 100 to 10,000 may be used as the plasticizer. As such a liquid resin, there may be mentioned a xylene resin, polybutadiene, polyoxypropylene glycol, polyoxypropylenetriol, a polyester resin, an acryl oligomer, NBR, SBR or polysulfide rubber.

Detailed Description Text (6):

The heat distortion resistance was determined by means of the peeling strength. After 24 hours, the heat distortion resistance was tested. For this purpose, the ASA film was loaded with a weight of 300 g at a peel-off angle of 180°. The temperature was increased by 10° C. every 30 minutes. The maximum temperature at which the peeling distance was just less than 50 mm was stated as the heat distortion resistance.

TABLE I

Heat distortion resistance in °C.	Example 4		Example 6		Example 8	
	4 × 60	4 × 60	4 × 60	4 × 60	4 × 80; 1 × 90	4 × 80; 1 × 90

Abbreviations

OHN=hydroxyl number

B14=1,4-butanediol

TDI=tolylene diisocyanate

HDI=hexamethylene diisocyanate

PUD=sodium salt of the Michael adduct of acrylic acid and ethylenediamine

DBTL=dibutyltin dilaurate

DMPA=dimethylolpropionic acid

FC=solids content

The viscosities of the dispersions were measured at 20° C. and a shear rate of 250s⁻¹ using a rotational rheometer having concentric cylinders (spindle diameter 38.7 mm, cup diameter: 42.0 mm).

The particle size of the latex particle (LD) was determined indirectly by means of turbidity measurements. For this purpose, the turbidity of a dispersion having a solids content of 0.01% by weight was determined relative to distilled water with a path length of 2.5 cm and at room temperature. The K value was determined by the method described in Kirk-Othmer, Encyclopedia of Chemical Technology, Third Edition, John Wiley & Sons, Inc. 1983, Volume 23, page 967. The solvent used was N-methylpyrrolidone.

We claim:

1. An aqueous polyurethane dispersion having latent crosslinking properties and containing

(I) a disperse phase (PI), containing

(Ia) a polyurethane (PUR.Ia) which, in addition to groups which impart water dispersibility to the polyurethane, carries groups which have a C—C double bond and in which the double bond is activated by a carbonyl group bonded directly therewith, or

(Ib) a mixture of

a polyurethane (PUR.Ib) which carries groups which impart water dispersibility to the polyurethane but not groups which have a C—C double bond and in which the double bond is activated by a carbonyl group bonded directly therewith and

a compound (VI) which differs from the polyurethanes PUR.Ia and PUR.Ib, and which carries groups which have a C—C double bond and in which the double bond is activated by a carbonyl group bonded directly therewith and

(II) a disperse phase (PII) containing a compound which differs from the compounds (PUR.Ia), (PUR.Ib) and (VI) and which carries a plurality of groups selected from the group consisting of the thiol groups, primary amino groups and secondary amino groups.

21.5 g of DMPA were reacted with 152.5 g of TDI for 2 hours at 110° C. After dilution with 977 g of acetone and cooling to 50° C., the NCO content was 0.5%. Neutralization was effected with 17.9 g of 25% strength NaOH and dispersing was carried out with 1200 g of demineralized water. Immediately after the end of dispersing, 46.9 g of pentaerythritol tetraakis-3-mercaptopropionate in 100 g of acetone were added dropwise. The acetone was distilled off under reduced pressure and the dispersion was brought to a solids content of 35%.
SH content: 0.24 mol/kg
Analytical data:
FC: 35%
LD: 97
Viscosity: 290 mPas
pH: 8.4
K value: 44

Example 6

572.3 g of a polyesterol obtained from adipic acid and ethylene glycol (OHN=42), 0.5 g of DBTL, 21.4 g of DMPA and 38.6 g of 1,4-butanediol were reacted with 167.7 g of TDI for 50 minutes at 110° C. Thereafter, dilution was effected with 977 g of acetone and the mixture was cooled to 50° C. The NCO value was now 0.67%. Neutralization was effected with 6.4 g of NaOH in 20 g of demineralized water and dispersing was carried out with 1200 g of demineralized water. Immediately thereafter, 78.2 g of pentaerythritol tetraakis-3-mercaptopropionate in 100 g of acetone were added dropwise. After the addition of 8 g of a 45% strength solution of polyacrylic acid (pH=7) in 30 g of water, the acetone was distilled off and the dispersion was brought to a solids content of 40%.

Analytical data:
Thiol content: 0.4 mol/kg

FC: 40%
LD: 98
Viscosity: 113 mPas
pH: 8.6

K value: undissolved

Preparation of the dispersions containing PIa and PII

Example 7

The dispersions from Example 1 and Example 5 were mixed in ratios such that equivalent amounts of SH and acrylate groups were present. K value: 45.6
A film was cast and was dried for 3 days at 40° C. K value: 50 undissolved particles.

Example 8

100 g of the dispersion from Example 4 and 181.2 g of the mixture: 44.3. The mixture was cast as a film, which was dried for 3 days at 40° C. K value of the film: undissolved. Testing of the performance characteristics of the dispersions were investigated by the following method:
The adhesive properties of the dispersions stated in Table 1 were investigated by the following method:
The dispersions were thickened with 2% of Collacral VL, each applied in a thickness of 2 mm to 5 hard particle boards (20 cm×3 cm) by means of a knife coater and dried for 3 minutes at 60° C. An ASA film was then pressed on at 0.5N/mm² for 30 seconds at 80° C.

Preparation of a sealant composition

Detailed Description Text (8):

Then, the above-mentioned operation was repeated to obtain two compositions having the same formulation. To the respective compositions, 1 part of the above-mentioned condensation product (b.sup.1) or (b.sup.2) as the partial addition condensation product (B) and 1 part of dibutyl tin acetate as the condensation catalyst (C) were added, and the mixtures were stirred for 30 minutes in a nitrogen stream, whereby 2 sealant compositions were obtained. These sealant compositions were put in sealed containers, respectively, and stored.

Detailed Description Text (10):

The compositions of the present invention prepared in the above-mentioned manner were respectively coated on an aluminum plate, a glass plate, a melamine-coated steel plate and a hard polyvinyl chloride resin sheet in a bead pattern having a thickness of 5 mm, a width of 10 mm and a length of 100 mm to obtain test pieces. These test pieces were cured at 20.degree. C. under a relative humidity of 65% for 7 days at room temperature.

Detailed Description Text (15):

Sealant compositions were prepared in the same manner as in Example 1 except that instead of the condensation product (b.sup.1) or (b.sup.2), .gamma.-aminopropyltriethoxysilane or Epikote 828.RTM. was incorporated. The adhesion and peeling tests were conducted in the same manner as in Example 1. The results are shown in Table 1.

Detailed Description Text (20):

Then, eight sealant compositions were prepared in the same manner as in Example 1 except that instead of the condensation product (b.sup.1) or (b.sup.2) used in Example 1, condensation products (b.sup.3) to (b.sup.10) of Table 2 were respectively used. The sealant compositions were subjected to the adhesion and peeling tests in the same manner as in Example 1. The results are shown in Table 3.

Detailed Description Text (22):

Sealant compositions were prepared in the same manner as in Example 1 except that instead of the condensation product (b.sup.1) or (b.sup.2) used in Example 1, N-.beta.(aminoethyl).gamma.-aminopropyltrimethoxysilane, .gamma.-glycidoxypropyltrimethoxysilane and .gamma.-methacryloxypropyltrimethoxysilane were respectively used. The sealant compositions were subjected to the adhesion and peeling tests in the same manner as in Example 1. The results are shown in Table 3.

Detailed Description Text (26):

Six sealant compositions were prepared in the same manner as in Example 1 except that instead of the condensation product (b.sup.1) or (b.sup.2) used in Example 1, condensation products (b.sup.11) to (b.sup.16) were respectively used. The sealant compositions were subjected to the adhesion and peeling tests as in Example 1. The results are shown in Table 5.

Detailed Description Text (31):

Two sealant compositions were prepared in the same manner as in Example 1 except that instead of the condensation product (b.sup.1) or (b.sup.2) used in Example 1, the condensation product (b.sup.17) or (b.sup.18) was used, respectively. The sealant compositions were subjected to the adhesion and peeling tests in the same manner as in Example 1. The results are shown in Table 6.

Detailed Description Text (34):

The representative sealant compositions prepared in Examples 1 to 4 were respectively coated on aluminum plate on a bead pattern having a thickness of 2 mm, a width of 10 mm and a length of 100 mm, and cured at 20.degree. C. under a relative humidity of 65% for 7 days. Then, the applied sealant compositions were subjected to irradiation of ultra-violet rays for 500 hours by means of a Sun Shine Weather Meter, whereupon the change of surface condition was observed. The results thereby obtained are shown in Table 7.

Detailed Description Text (36):

The representative sealant compositions prepared in Examples 1 to 4 and placed in sealed containers, were subjected to accelerated storage at 50.degree. C. for 30 days. Then, they were brought to 20.degree. C., and their viscosities were measured by a Brook Field Type Rotary Viscometer. The test results are shown in Table 7.

2. An aqueous dispersion as claimed in claim 1, wherein the phase (P1) contains a polyurethane (PUR.1a) composed of (a1) polyfunctional isocyanates of 4 to 30 carbon atoms, (a2) polyols, of which a2.1 from 10 to 100 mol %, based on the total amount of the diols (a2), have a molecular weight of from 500 to 5000 and a2.2 from 0 to 90 mol %, based on the total amount of the diols (a2), have a molecular weight of from 500 g/mol, (a3) monomers which differ from the monomers (a1) and (a2) and carry at least one isocyanate group or at least one group reactive toward isocyanate groups and furthermore carry at least one hydrophilic group or one potentially hydrophilic group, imparting water-dispersibility to the polyurethanes, (a4) monomers which differ from the monomers (a1), (a2) and (a3) and carry at least one isocyanate group or at least one group reactive toward isocyanate groups which furthermore carry at least one acryloyl or methacryloyl group and (a5) if required, further polyfunctional compounds which differ from the monomers (a2) to (a4) and have groups which are reactive toward isocyanate groups and which are alcoholic hydroxyl groups or primary or secondary amino groups.

3. An aqueous dispersion as claimed in claim 1, wherein the phase (P1) is a mixture of a polyurethane (PUR.1b) and a polyurethane (PUR.1c) as compound (V1), the polyurethane (PUR.1b) being composed of (b1) polyfunctional isocyanates of 4 to 30 carbon atoms, of which b2.1 from 10 to 100 mol %, based on the total amount of the diols (b2), have a molecular weight of from 500 to 5000 and b2.2 from 0 to 90 mol %, based on the total amount of the diols (b) have a molecular weight of from 500 g/mol, (b3) monomers which differ from the monomers (b1) and (b2) and have at least one isocyanate group or at least one group reactive toward isocyanate groups and furthermore carry at least one hydrophilic group or one potentially hydrophilic group imparting water-dispersibility to the polyurethanes, (b4) if required, further polyfunctional compounds which differ from the polyols (b2) and monomers (b3) and have groups which are reactive toward isocyanate and are alcoholic hydroxyl groups or primary or secondary amino groups, (c1) polyfunctional isocyanates of 4 to 30 carbon atoms, of which c2.1 from 10 to 100 mol %, based on the total amount of the diols (c2), have a molecular weight of from 500 to 5000 and c2.2 from 0 to 90 mol %, based on the total amount of the diols (c2), have a molecular weight of from 500 g/mol.

(c3) and monomers which differ from the monomers (c1) and (c2) and have at least one isocyanate group or at least one group reactive toward isocyanate groups and furthermore carry at least one acryloyl or methacryloyl group.

4. An aqueous dispersion as claimed in claim 3, wherein the monomer (a4) or (c3) is a hydroxy-C₁-C₆-alkyl acrylate, a hydroxy-C₁-C₆-alkyl methacrylate or the bisadduct of an acrylic acid or methacrylic acid with a bisepoxide.

5. An aqueous dispersion as claimed in claim 1, wherein the phase (P1) contains a polyurethane (PUR.1d) which carries groups which impart water dispersibility to the polyurethane, a polymer (POL.1a) prepared from monomers capable of free radical polymerization and having C=C double bonds by the method of free radical emission or suspension polymerization or a polymer (POL.1d) which carries groups which impart water dispersibility to the polymer, prepared from monomers capable of free radical polymerization and having C=C double bonds by the method of free radical mass or solution polymerization, the polyurethane (PUR.1e) or the polymers (POL.1d) or (POL.1e) carrying less than 0.05 mol of primary amino groups, secondary amino groups or thiol groups per kg.

6. An aqueous dispersion as claimed in claim 1, wherein the phase (P1) contains a mixture of a polyurethane (PUR.1e) which carries groups which impart water dispersibility to the polyurethane, a polymer (POL.1e) prepared from monomers capable of free radical polymerization and having C=C double bonds by the method of free radical emission or suspension polymerization or a polymer (POL.1d) which carries groups which impart water dispersibility to the polymer, prepared from monomers capable of free radical polymerization and having C=C double bonds by the method of free radical mass or solution polymerization, the polyurethane (PUR.1e) or the polymers (POL.1d) or (POL.1e) carrying less than 0.05 mol of primary amino groups, secondary amino groups or thiol groups per kg, and a compound (VII) which carries at least 2 groups selected from the group consisting of the thiol groups, primary amino groups or secondary amino groups and has a weight of less than 1 g/l in water and a molecular weight of less than 1000.

7. An aqueous dispersion as claimed in claim 6, wherein the compound (VII) is an araliphatic C₆-C₃₀-polyamine or an aliphatic C₄-C₃₀-polyamine or an ester synthesized from a polyhydric C₂-C₁₀-alkyl alcohol and a C₂-C₁₀-thiomonocarboxylic acid.

8. An aqueous dispersion as claimed in claim 7, wherein the compound (VII) is pentaerythritol tetraakis-3-mercaptopropionate.

9. An article of wood, metal, textile, leather or plastic which is adhesively bonded, impregnated or coated with an aqueous dispersion as claimed in claim 1.

10. A method of coating or adhesively bonding a substrate comprising applying to said substrate an aqueous dispersion as claimed in claim 1.

CLAIMS:

1. A one component room temperature curable sealant composition comprising:

(A) a polyether polymer having at its terminal a hydrolyzable silicon functional group represented by the formula: ##STR15## wherein R.sup.I is a monovalent hydrocarbon group having from 1 to 12 carbon atoms, R.sup.II is a monovalent hydrocarbon group having from 1 to 6 carbon atoms, and n is an integer of 0 to 2;

(B) a partial addition condensation product of:

(a) an aminoalkylalkoxysilane represented by the formula: ##STR16## wherein R.sup.1 is a divalent hydrocarbon group having from 1 to 4 carbon atoms, each of R.sup.2 and R.sup.3 is a monovalent hydrocarbon group having from 1 to 4 carbon atoms, Z is a hydrogen atom or an aminoalkyl group, and a is an integer of 0 or 1, with

(b) an epoxy compound containing at least one epoxy group in its molecular structure and having a molecular weight of from 100 to 1,000 and an epoxy equivalent of from 100 to 500, wherein 0.3-1.0 equivalents of epoxy are used per equivalent of aminoalkylalkoxysilane,

(c) a compound selected from the group consisting of .gamma.-glycidoxypropyltrimethoxysilane, .gamma.-glycidoxypropylmethyldiethoxysilane, 3,4-epoxycyclohexylethyltrimethoxysilane, .gamma.-methacryloxypropyltrimethoxysilane .gamma.-methacryloxypropylmethyldimethoxysilane,

(d) an alkoxysilane selected from the group consisting of vinyltrimethoxysilane, vinyltriethoxysilane, .gamma.-mercaptopropyltrimethoxysilane, .gamma.-chloropropyltrimethoxysilane, vinyl tris (.beta.-methoxyethoxy) silane, dimethyl dimethoxysilane, methyltrimethoxysilane, tetramethoxysilane, dimethyldimethoxysilane, methyltriethoxysilane, tetraethoxysilane, diphenyldimethoxysilane, and phenyltrimethoxysilane; and

(e) an organic titanate acid ester; or a mixture thereof, and

(C) a condensation catalyst of a silanol compound.

2. The one component room temperature curable sealant composition according to claim 1, which comprises 100 parts by weight of the polyether polymer (A), from 0.5 to 10 parts by weight of the partial addition condensation product (B) and from 0.05 to 5 parts by weight of the condensation catalyst (C).

3. The one component room temperature curable sealant composition according to claim 1, wherein the partial addition condensation product is a condensation product of one equivalent of the aminoalkylalkoxysilane (a) with from 0.3 to 1.0 equivalent of the epoxy compound (b), from 0.03 to 2.6 equivalent of the compound (c), from 0.03 to 2.6 equivalent of the alkoxysilane (d), and from 0.1 to 1.2 equivalent of the organic titanate ester (e) or a mixture thereof.

4. The one component room temperature curable sealant composition according to claim 1, wherein the polyether polymer (A) has a principal chain comprising repeating units represented by the general formula --R--O-- where R is a divalent alkylene group having from 2 to 4 carbon atoms.

5. The one component room temperature curable sealant composition according to claim 1, wherein the polyether polymer (A) is a reaction product of a polyether urethane prepolymer having a terminal isocyanate group and .gamma.-aminopropyltrimethoxysilane, .gamma.-aminopropyltriethoxysilane or .gamma.-mercaptopropyltrimethoxysilane, or an oxypropylene polymer having a terminal silyl ether group represented by the formula: ##STR17##

6. The one component room temperature curable sealant composition according to claim 1, wherein the aminoalkylalkoxysilane (a) is selected from the group consisting of aminomethyltriethoxysilane, N-.beta.-(aminoethyl) aminomethyltrimethoxysilane, aminomethyldiethoxysilane, N-.beta.-(aminoethyl) methyltributoxysilane, .gamma.-aminopropyltrimethoxysilane, .gamma.-aminopropyltriethoxysilane, .gamma.-aminopropylmethyldiethoxysilane, .gamma.-aminoisobutyltrimethoxysilane, N-.beta.-(aminoethyl).gamma.-aminopropyltrimethoxysilane, N-.beta.-(aminoethyl)

United States Patent [19]

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Jan. 31, 1984

Saito et al.

Best Available Copy [45]

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Encyclopedia of Polymer Science and Technology, vol. 13, pp. 396-397.

Primary Examiner—J. Ziegler

Attorney, Agent, or Firm—Birch, Stewart, Kolasch &

Birch

ABSTRACT

A thermoplastic polymer composition containing (i) a modified block copolymer of an aromatic vinyl compound and a conjugated diene compound, which is modified with a dicarboxylic acid or the derivative thereof, or the ionically crosslinked product of the modified block copolymer with a univalent, bivalent or trivalent metal ion and (ii) a thermoplastic polymer having a polar group. This thermoplastic polymer composition has excellent mechanical and other properties. The compatibility of each component in the thermoplastic composition is remarkably improved.

49 Claims, No Drawings

[54] THERMOPLASTIC POLYMER COMPOSITION

[56]

[75] Inventors: Akira Saito, Fujisawa; Akio Yamori, Kawasaki; Hideo Morita, Yokohama, all of Japan
[73] Assignee: Asahi Kasei Kogyo Kabushiki Kaisha, Osaka, Japan

[21] Appl. No.: 157,167

[22] Filed: Jun. 6, 1980

[30] Foreign Application Priority Data

Jun. 13, 1979	[JP]	Japan	54-73492
Feb. 16, 1980	[JP]	Japan	55-17210
Feb. 16, 1980	[JP]	Japan	55-17212
Feb. 16, 1980	[JP]	Japan	55-17213
Feb. 16, 1980	[JP]	Japan	55-17214
Feb. 16, 1980	[JP]	Japan	55-17215
Feb. 22, 1980	[JP]	Japan	55-20568
Feb. 22, 1980	[JP]	Japan	55-20582
C08L 29/04			
525/57; 525/63; 525/78; 525/80; 525/88; 525/92; 525/94; 525/301	Field of Search		
525/78, 80, 88, 92, 94			

Set aside

8, 11, 14

.gamma.-aminopropylmethyldimethoxysilane, and [{N-.beta.(aminoethyl)}
N-.beta.(amonoethyl)] .gamma.-aminopropyltrimethoxysilane.

7. The one component room temperature curable sealant composition according to claim 1, wherein the epoxy compound (b) is a bisphenol A type epoxy compound.

8. The one component room temperature curable sealant composition according to claim 1, wherein the organic titanate acid ester is selected from the group consisting of tetraisopropyl titanate tetra-n-butyl titanate, butyl titanate dimer and tetra (2-ethylhexyl) titanate.

9. The one component room temperature curable sealant composition according to claim 1, wherein the condensation catalyst (C) of a silanol compound is selected from the group consisting of an organic silicone titanate, stannous octoate dibutyl tin diacetate, dibutyl tin dilaurate and dibutylamine-2-ethylhexoate.

THERMOPLASTIC POLYMER COMPOSITION

The present invention relates to a novel thermoplastic polymer composition. More specifically, it relates to a thermoplastic polymer composition containing (i) a modified block copolymer of an aromatic vinyl compound and a conjugated diene compound, which is modified with a dicarboxylic acid or the derivative thereof, or the ionically crosslinked product thereof and (ii) a thermoplastic polymer having polar groups containing polar atoms, such as oxygen, nitrogen, sulfur and halogen atoms and halogen atoms and, optionally, (iii) styrene polymers of polyolefins. This thermoplastic polymer composition has excellent properties including mechanical properties and the compatibility of each component in the thermoplastic polymer composition is remarkably improved.

Various polymer substances have heretofore been used as fibers, films, sheets, molded articles and the like. However, desired products having a desired property or properties cannot be obtained by the use of a single polymer substance. For this reason, various attempts to improve the processability of products, to adjust the balance of the physical properties of products or to lower the cost of products have been made by, for example, the combination of two or more polymer substances having different properties, the mixing or blending of various polymer substances together with low-molecular-weight substances or inorganic substances, or the laminating of two or more layers. However, in the case where various polymeric substances are mixed to prepare compositions, the compatibility of the different polymer substances to be mixed is not necessarily good. As a result, in many cases, the desired modification of certain polymer substances by these attempts cannot be readily obtained due to, for example, the non-uniformity or non-homogeneity and the delamination, based on the poor intermixing properties and compatibility of aromatic vinyl components and conjugated diene compounds, typically including styrene-butadiene block copolymers, are one of the polymer substances which are recently noted in the art. Among the styrene-butadiene block copolymers, those containing two or more polystyrene blocks and one or more polybutadiene blocks and having a relatively small styrene content have rubber elasticity similar to that of conventional vulcanized rubbers and also have moldability of processability similar to those of conventional thermoplastic plastic materials. Therefore, these styrene-butadiene block copolymers are widely used in the field of, for example, molding materials, such as soles of shoes, the modification of polystyrene materials (for shoes), the modification of polystyrene resins and the like, adhesives, bonding agents and the like. On the other hand, styrene-butadiene block copolymers having a high styrene content are used, as a clear high-impact styrene resin, in the field of, for example, packaging materials.

The block copolymers of aromatic vinyl compounds and conjugated diene compounds are useful by themselves and, also when they are mixed with, for example, styrene polymers and polyolefins, provide useful polymer compositions having desired properties. However, since these block copolymers are composed of only hydrocarbon monomers and since they are not compatible with other thermoplastic polymer substances, especially those containing a polar group in which a polar atom such as oxygen, nitrogen, sulfur or halogen atoms is contained, useful polymer compositions having desired properties cannot be obtained when these block copolymers are mixed with the above-mentioned other thermoplastic polymer substances.

Accordingly, an object of the present invention is to obviate the above-mentioned problems of the prior art and to provide a thermoplastic polymer composition containing the block copolymer of the aromatic vinyl compound and the conjugated diene compound, in which the compatibility of the block copolymer with a thermoplastic polymer substance containing a polar group is improved and the properties, such as the impact resistance and the heat resistance thereof, are improved. Other objects and advantages of the present invention will be apparent from the following description.

In accordance with the present invention, there is provided a thermoplastic polymer composition comprising:

(a) 1 through 99 parts by weight of a component A consisting essentially of at least one member selected from the group consisting of modified block copolymers and the ionically crosslinked products of at least one said modified block copolymer with at least one univalent, bivalent or trivalent metal ion, said modified block copolymer comprising a block copolymer of at least one aromatic vinyl compound and at least one conjugated diene compound onto which at least one molecular unit containing at least one dicarboxylic acid group or the derivative thereof is grafted; and

(b) 99 through 1 parts by weight of a component B consisting essentially of at least one thermoplastic polymer having polar groups.

The above-mentioned thermoplastic polymers containing polar groups of the component B include polyamides, polyurethanes, vinylalcohol polymers, polyacrylates, polymethacrylates, chlorinated hydrocarbon polymers, ionomers and oligomers other than the above-mentioned polymers, said oligomers having at least one polar group which is reactive to the dicarboxylic acid or the derivative thereof and having a number-average molecular weight of 100 through 10,000.

In addition to the components A and B, the thermoplastic polymer composition of the present invention may optionally contain 0 through 100 parts by weight of at least one member selected from the group consisting of styrene polymers and polyolefins, based on 100 parts by weight of the total amount of the components A and B.

According to the present invention, since the modified block copolymers containing dicarboxylic acid groups or the derivatives thereof are used as the component A, the interaction of the component A with respect to the various thermoplastic polymers containing polar groups used as the component B is increased as, compared to the unmodified block copolymer. As a result, the mechanical and chemical properties of the present thermoplastic polymer compositions containing the components A and B are improved, compared to those of the compositions containing the unmodified block copolymers. Especially, when the thermoplastic polymers containing polar groups such as an amino group, hydroxyl group, epoxy group and isocyanate group, which can be reacted or interacted with the dicarboxylic acid groups or the derivatives thereof contained in the modified block copolymers or the ionically crosslinked dicarbox-

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Search Results - Record(s) 1 through 5 of 5 returned.

☐ 1. Document ID: US 6274693 B1

L2: Entry 1 of 5

File: USPT

Aug 14, 2001

US-PAT-NO: 6274693

DOCUMENT-IDENTIFIER: US 6274693 B1

TITLE: Method for producing a two-layer coating and suitable enamel therefor

DATE-ISSUED: August 14, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Poth; Ulrich	Munster			DE
Wiatr; Barbel	Munster			DE

US-CL-CURRENT: 528/45; 427/385.5, 427/407.1, 544/224, 560/158

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

☐ 2. Document ID: US 6060573 A

L2: Entry 2 of 5

File: USPT

May 9, 2000

US-PAT-NO: 6060573

DOCUMENT-IDENTIFIER: US 6060573 A*König*

TITLE: Polyisocyanates blocked with CH-acidic esters and stabilized against thermal yellowing

DATE-ISSUED: May 9, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
König; Eberhard	Leverkusen			DE
Kobusch; Claus	Meerbusch			DE
Kuttner; Wolfram	Bergisch Gladbach			DE

US-CL-CURRENT: 528/45; 252/182.2, 524/770, 524/772, 528/73, 560/158, 560/331

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Image								

KWC

☐ 3. Document ID: US 5039720 A

and fumaric acid. Otherwise, these polyesterers have the same composition as the polyesterers which are suitable as monomers (a2.2).

The monomers (a5) which may be used as components and differ from the monomers (a2) to (a4) are used in general for crosslinking or chain extension. They are in general trihydric or polyhydric nonaromatic alcohols, amines having 2 or more primary and/or secondary amino groups and compounds which carry one or more primary and/or secondary amino groups in addition to one or more alcoholic hydroxyl groups. Alcohols which have a functionality of more than 2 and may be used for establishing a certain degree of branching or crosslinking are, for example, trimethylolpropane, glycerol and sugar.

Monocarboxylic acids, in addition to the hydroxyl group, carry a further group reactive toward isocyanates, such as monocarboxylic acids having one or more primary and/or secondary amino groups, eg. monoethanolamine, are also suitable. Polyamines having 2 or more primary and/or secondary amino groups are used particularly when the chain extension or crosslinking is to be carried out in the presence of water, since amines generally react more rapidly than alcohols or urethanes having a high molecular weight are desired. In such cases, the prepolymer having isocyanate groups is prepared, rapidly dispersed in water and then subjected to chain extension or crosslinking by adding compounds having a plurality of amino groups reactive toward isocyanates. Amines suitable for this purpose are in general polyfunctional amines having a molecular weight of from 32 to 500, preferably from 60 to 300, g/mol, which contain at least two primary amino groups, two secondary amino groups or one primary and one secondary amino group. Examples of these diamines are diaminobutane, diaminopropane, diaminobenzene, diaminodiphenylmethane, 2,5-dimethylpiperazine, amino-3-aminomethyl-3,5,5-trimethylcyclohexane (isophoronediamine, IPDA), 4,4'-diaminodicyclohexylmethane, 1,4-diaminocyclohexane, aminocyclohexanediol, hydrazine and hydrazine hydrate, and triamines, such as diethylenetriamine and 1,8-diaminotetraamine.

The amines may also be used in blocked form, for example in the form of the corresponding ketimines (cf. for example CA-1-129 128), ketazimes (cf. for example U.S. Pat. No. 4,269,748) or amine salts (cf. U.S. Pat. No. 4,192,937). Oxazolidines, as for example in U.S. Pat. No. 4,192,937, are also block polyamines which may be used in the preparation of the novel polyurethanes for chain extension of the prepolymer. When such blocked polyamines are used, they are generally mixed with the dispersing water in the absence of water and the mixture is then mixed with the dispersing water or a part of the dispersing water so that the corresponding polyamines are liberated hydrolytically. Mixtures of di- and triamines are preferably used, particularly preferably mixtures of isophoronediamine and diethylenetriamine.

The polyurethanes preferably contain no polyamine or from 1 to 10, particularly preferably from 4 to 8, mol %, based on the total amount of component (a2) to (a4), of a polyamine having at least 2 amino groups reactive toward isocyanates, as monomers (a5).

It is advisable to use compounds having primary or secondary amino groups only in amounts such that the total amount of the isocyanate groups present at the time of the addition is greater than that of the primary or secondary amino groups.

55 The disperse phase (P1) may also contain, as compounds (VI), acryloyl- or methacryloyl-containing esters disclosed in EP-A-447 845, 279 303 or 127 766.

These are preferably esters obtainable by reacting polyols as described as monomers (a2.1) and (a2.2), and low molecular weight higher alcohols, such as glycerol, trimethylolpropane and pentaerythritol, these alcohols being, if required, ethoxylated or propoxylated, dibasic to tetrabasic C₃-C₉-carboxylic acids, eg. adipic acid, and acrylic and/or methacrylic acid, to give a carboxyl-containing polyester, and then esterifying the carboxyl groups of this ester by reaction with an equivalent amount of an epoxide compound.

L2: Entry 3 of 5

File: USPT

Aug 13, 1991

US-PAT-NO: 5039720

DOCUMENT-IDENTIFIER: US 5039720 A

TITLE: Aqueous electrophoretic enamel coating materials, which can be deposited at the cathode crosslinked with methane tricarboxylic acid amides of malonic acid derivatives

DATE-ISSUED: August 13, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Saatweber; Dietrich	Wuppertal			DE
Hendriux; Georg	Wuppertal			DE
Brindopke; Gerhard	Sulzbach/Ts			DE
Plum; Helmut	Taunusstein			DE

US-CL-CURRENT: 523/404; 523/407, 523/408, 523/410, 523/414, 523/415, 523/417, 523/420, 525/111, 525/113, 525/504, 528/119, 528/120, 528/45, 528/73

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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KWIC

☐ 4. Document ID: JP 07207223 A

L2: Entry 4 of 5

File: JPAB

Aug 8, 1995

PUB-NO: JP407207223A

DOCUMENT-IDENTIFIER: JP 07207223 A

TITLE: THERMOSETTING COATING COMPOSITION

PUBN-DATE: August 8, 1995

INVENTOR-INFORMATION:

NAME	COUNTRY
MORIYA, YOICHI	
MARUYAMA, CHIKASHI	
SHIBAFUJI, KISHIO	
PEETAA, BETSUTSU	

INT-CL (IPC): C09 D 175/04; C09 D 161/32; C08 F 290/06; C08 G 18/62

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
Draw Desc	Clip Img	Image							

KWIC

☐ 5. Document ID: DE 2342603 A BE 819042 A DE 2342603 B DK 7404494 A FR 2241573 A GB 1442024 A IT 1018973 B NL 7411118 A SE 7410681 A

L2: Entry 5 of 5

File: DWPI

Mar 6, 1975

DERWENT-ACC-NO: 1975-17772W

DERWENT-WEEK: 197511

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TITLE: Lacquer soluble isocyanate-contg. addtn. prods. - of malonic acid dialkyl esters

70100
70100

and aliphatic biuret polyisocyanates

PRIORITY-DATA: 1973DE-2342603 (August 23, 1973)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE 2342603 A	March 6, 1975		000	
BE 819042 A	February 21, 1975		000	
DE 2342603 B	October 16, 1980		000	
DK 7404494 A	April 28, 1975		000	
FR 2241573 A	April 25, 1975		000	
GB 1442024 A	July 7, 1976		000	
IT 1018973 B	October 20, 1977		000	
NL 7411118 A	February 25, 1975		000	
SE 7410681 A	March 24, 1975		000	

INT-CL (IPC): C07C 119/04; C07C 127/24; C08F 0/00; C08G 18/72; C09D 3/72

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments
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Term	Documents
POLYISOCYANATE.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	52877
POLYISOCYANATES.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	22356
MALONIC.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	27243
MALONICS	0
ESTER.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	475265
ESTERS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	300462
((MALONIC NEAR3 POLYISOCYANATE) ADJ ESTER).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	5
(POLYISOCYANATE NEAR3 MALONIC ESTER).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	5

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Other suitable monomers (d4) are aminoacids and alcohols containing thiol groups, eg. $\text{CH}_2\text{SH}-\text{CH}_2\text{OH}$ or $\text{CH}_2\text{OH}-\text{CHOH}-\text{CH}_2\text{SH}$.

In the preparation of the dispersion (D.II) which contains, as the polyurethane (PUR.IId), one having amino groups as functional groups, the prepolymer mixing process is adapted in the usual manner, the prepolymer is dispersed in water and the monomer (d4) is then added to the dispersion. The amount of monomer (d4) is preferably chosen so that in the prepolymer is from 0.9:1 to 1.1:1.

Furthermore, dispersions which contain a polyurethane which carries primary and/or secondary amino groups are disclosed in DE-A-2 734 576, DE-A-2 753 942, EP-A-390 370 and EP-A-442 654.

Dispersions (D.II) containing polyurethanes (PUR.IId) having thiol groups are disclosed, for example in DE-A-2 642 073.

These water-insoluble compounds preferably have a water solubility of less than 5, particularly less than 1, g/l in water at 20° C.

Suitable dispersions (D.II) which contain a polymer (POL.IIa) as the disperse phase are primary dispersions capable of free radical polymerization are polymerized by the method of suspension or emulsion polymerization in an aqueous medium. Isocyanates having double bonds capable of free radical polymerization (for example TMXD) or thiol suitable as regulators, as disclosed in U.S. Pat. No. 5,227,463, are used for functionalizing the polymers (POL.IIa) having primary amino groups, secondary amino groups or thiol groups.

Other suitable dispersions (D.II) which contain a polymer (POL.IIb) as the disperse phase are secondary dispersions containing (POL.IId) which carries groups which impart water dispersibility to the polymer, eg. carboxyl groups. These dispersions are prepared from conventional monomers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds in the presence of acrylic acid and/or methacrylic acid by the method of free radical mass or solution polymerization and are then dispersed in water. For subsequent functionalization with primary amino groups, these dispersions can be reacted with ethylenimine, as stated in EP-A-341 886.

In an embodiment which is likewise preferred, the dispersion (D.II) used is a dispersion in which the disperse phase (II) contains or preferably consists of a mixture of a polyurethane (PUR.IIe) which particularly preferably has the same composition as the polyurethane (PUR.Ib) and a compound (VII). The process for the preparation of these dispersions is similar to that for the preparation of dispersions (D.I) which contain a mixture of polyurethane (PUR.Ib) and compound (VI) as the disperse phase, ie. a mixture of the polyurethane (PUR.IIe) or the prepolymer thereof and the compound (VII) is dispersed in water.

Suitable dispersions (D.II) are those in which the disperse phase (II) contains or preferably consists of a mixture of a polymer (POL.IId) prepared from monomers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds by the method of free radical mass or solution polymerization. These are in general secondary dispersions which are obtainable by polymerizing, by the mass or solution polymerization method, conventional monomers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds, a part of these monomers carrying hydrophilic groups, eg. carboxyl groups. The compound (VII) is added after the end

The phase (P.II) preferably contains or particularly preferably consists of a polyurethane (PUR.IId) which carries groups which impart water dispersibility to the polyurethane, a polymer (POL.IIa) prepared from monomers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds by the method of free radical mass or solution polymerization, or a polymer (POL.IId) which carries groups which impart water dispersibility to the polymer, said polymer being prepared from polymers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds by the method of free radical mass or solution polymerization. The polyurethane (PUR.IIa) or the polymer (POL.IIa) or (POL.IIb) preferably carries from 50 to 1000 mmol/kg of primary amino groups, secondary amino groups or thiol groups.

Preferably both the polyurethane (PUR.IId) and the polymer (POL.IIa) (POL.IIb) have a solubility of less than 5, particularly preferably less than 1, g/l in water (measured at 20° C.).

In an embodiment which is likewise preferred, the phase (P.II) consists of a mixture of

a polyurethane (PUR.IIe) which carries groups which impart water dispersibility to the polyurethane, a polymer (POL.IIc) prepared from monomers capable of free radical polymerization and having $\text{C}=\text{C}$ double bonds by the method of free radical polymerization and having $\text{C}=\text{C}$ double bonds by the method of free radical mass or solution polymerization, the polyurethane (PUR.IIa) or the polymer (POL.IIc) or (POL.IId) carrying less than 0.05 mmol of primary amino groups, secondary amino groups or thiol groups per kg, and

a compound (VII) which carries at least 2 groups selected from the group consisting of the thiol groups, primary amino groups and secondary amino groups and has a solubility of 1 g/l in water and a molecular weight of less than 1000.

Dispersions (D.II) which contain these disperse phases (P.II) and are free of disperse phases (P.I) and give the novel aqueous dispersions as a result of being mixed with dispersions (D.I) are generally known.

A preferably used dispersion (D.II) is a dispersion which contains, as the disperse phase (P.II), a polyurethane (PUR.IId) which is composed of

(d2) polyols of which

(d2.1) from 10 to 100 mol %, based on the total amount of the diols (d2), have a molecular weight of from 500 to 5000 and

(d2.2) from 0 to 90 mol %, based on the total amount of the diols (d2), have a molecular weight of from 60 to 500 g/mol,

(d3) monomers which differ from the monomers (d1) and (d2) and have at least one isocyanate group or at least one group reactive toward isocyanate groups and in addition carry at least one hydrophilic group or one potentially hydrophilic group, imparting water dispersibility to the polyurethanes, and

(d4) compounds which differ from the monomers (d2) and (d3) and have a plurality of groups which are primarily amino groups, secondary amino groups or thiol groups. Particularly suitable monomers (d1) to (d3) are the corresponding monomers which are preferred as monomers (a1) to (a3). Suitable monomers (d4) are the polyfunctional amines which are mentioned as monomers (a5).

WEST[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 4 of 4 returned.**☐ 1. Document ID: US 6040009 A

L5: Entry 1 of 4

File: USPT

Mar 21, 2000

US-PAT-NO: 6040009

DOCUMENT-IDENTIFIER: US 6040009 A

TITLE: Low solvent content type-resin composition, coating composition containing such resin composition and process for coating such coating composition

DATE-ISSUED: March 21, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Marutani; Yoshiaki	Hiroshima			JP
Tomita; Takashi	Hiroshima			JP
Nakahama; Tadamitsu	Hiroshima			JP
Uemura; Hiroyuki	Hiroshima			JP
Koga; Kazuhi	Hiroshima			JP
Ohsawa; Mika	Hiroshima			JP

US-CL-CURRENT: 427/241; 427/240, 427/385.5, 427/388.2, 427/388.3, 523/400, 524/539, 524/542, 524/597, 525/437, 525/440, 525/443, 525/450, 525/519, 528/297, 528/354, 528/80, 528/81

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
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☐ 2. Document ID: US 5393823 A

L5: Entry 2 of 4

File: USPT

Feb 28, 1995

US-PAT-NO: 5393823

DOCUMENT-IDENTIFIER: US 5393823 A

TITLE: Coating resin composition

DATE-ISSUED: February 28, 1995

INVENTOR-INFORMATION:

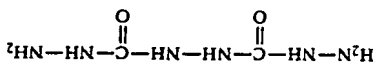
NAME	CITY	STATE	ZIP CODE	COUNTRY
Konno; Hidetoshi	Izumi-ohtsu			JP
Ooka; Masataka	Nara			JP
Takeda; Hirofumi	Yao			JP
Sakamoto; Hiroshi	Ikaruga			JP

US-CL-CURRENT: 524/507; 524/512, 524/535, 525/123, 525/160, 525/326.5, 525/355, 525/366, 525/370, 525/483, 526/229, 528/403

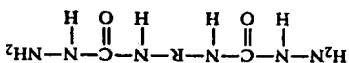
of a 40% strength aqueous sodium lauryl-sulfate solution in 5,000 parts of water. After completion of the polymerization, the pH is brought to 7 with ammonia. 47 parts of toluylene-bis-semicarbazide and 0.28 part of chromium sulfate are then stirred in. The dispersion may be used for coating paper. Even after several weeks' storage at room temperature, no free hydrazine is detectable. A film prepared from the dispersion picks up 540% of solvent after 1 day's storage in dimethyl-formamide.

EXAMPLE 15

1,000 parts of vinyl acetate, 400 parts of polyvinyl alcohol (88% hydrolyzed, viscosity of the 40% strength solution: 25 mPa.s), 200 parts of nonylphenol oxyethylated with 20 moles of ethylene oxide, 20 parts of sodium acetate and 6,500 parts of water are introduced into an autoclave equipped with a stirrer and thermostatic control. The charge is then heated to 55° C. and ethylene is forced in up to a pressure of 40 bar. This pressure is maintained during the subsequent polymerization. The polymerization is started by means of a solution of 40 parts of potassium persulfate in 2,000 parts of water and a solution of 40 parts of sodium bisulfite in 2,000 parts of water. In addition, a mixture of 7,000 parts of vinyl acetate and 500 parts of acryloxypropionaldehyde is introduced into the autoclave continuously over 3 hours. On completion of the addition, the polymerization is continued for 1 hour at 55° C. The reactor is then cooled, the pressure is let down and the dispersion is run off. Analysis shows a content of polymerized ethylene units of 15%. 250 parts of bis-semicarbazide of the formula



groups, which dispersion contains as a crosslinking agent for the keto and/or aldehyde groups of the copolymer from 0.02 to 1 mole of an organic hydrazine derivative from the group consisting of dihydrazides of aliphatic dicarboxylic acids of 2 to 10 carbon atoms and bis-semicarbazides of the general formula



in which R is an aliphatic radical of 2 to 7 carbon atoms or a carbocyclic radical of 6 to 8 carbon atoms, and from 0.0002 to 0.02 mole of heavy metal ions per mole of the hydrazine derivative.
2. The dispersion of claim 1 which contains from 0.0002 to 0.02 mole of copper, iron, manganese, vanadium, zinc, lead and/or cobalt ions per mole of a dihydrazide of an aliphatic dicarboxylic acid of 2 to 10 carbon atoms.

* * * * *

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC
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☐ 3. Document ID: US 4972027 A

L5: Entry 3 of 4

File: USPT

Nov 20, 1990

US-PAT-NO: 4972027

DOCUMENT-IDENTIFIER: US 4972027 A

TITLE: Composition curable at low temperature

DATE-ISSUED: November 20, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Isozaki; Osamu	Yokohama			JP
Nakai; Noboru	Hiratsuka			JP
Sugiura; Shinji	Fujisawa			JP
Ito; Satoru	Kanagawa			JP
Takami; Seiji	Hiratsuka			JP

US-CL-CURRENT: 525/365; 525/286, 525/288, 525/364, 525/72, 525/74

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 4. Document ID: US 4923945 A

L5: Entry 4 of 4

File: USPT

May 8, 1990

US-PAT-NO: 4923945

DOCUMENT-IDENTIFIER: US 4923945 A

TITLE: Resin composition curable at low temperature

DATE-ISSUED: May 8, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Isozaki; Osamu	Yokohama			JP
Nakai; Noboru	Hiratsuka			JP
Ito; Satoru	Kanagawa			JP
Takami; Seiji	Hiratsuka			JP

US-CL-CURRENT: 528/16; 524/199, 524/284, 524/285, 524/361, 524/366, 524/367, 524/376,
525/476, 525/477, 526/279, 528/17 , 528/32

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									



US005916960A

United States Patent [19]
Lum et al. [45]
Patent Number: 5,916,960
Date of Patent: *Jun. 29, 1999

[54] WATER-BASED, STORAGE STABLE, SELF-CURING COATING COMPOSITION

[75] Inventors: Dorey Lum, Suffern, N.Y.; Prashant Patel, Aliso Viejo, Calif.

[73] Assignee: The Sherwin-Williams Company, Cleveland, Ohio

[*] Notice: This patent is subject to a terminal disclaimer.

[21] Appl. No.: 08/955,418

[22] Filed: Oct. 22, 1997

Related U.S. Application Data

[63] Continuation of application No. 08/660,717, Jun. 10, 1996, abandoned, which is a continuation of application No. 08/171,577, Dec. 21, 1993, abandoned.

[51] Int. Cl.⁶ C08J 3/03; C08L 75/00
[52] U.S. Cl. 524/501, 524/548; 524/517

[58] Field of Search 524/501, 507, 524/548, 517

[56] References Cited

3,862,074 1/1975 Hickey 260/29.6
4,066,591 1/1978 Scrives et al. 260/29.2
4,108,811 8/1978 Eckhoff 260/22

U.S. PATENT DOCUMENTS

FOREIGN PATENT DOCUMENTS

0350040 1/1990 European Pat. Off.
3440535 5/1986 Germany.
57-61050 4/1982 Japan.
2-91170 3/1990 Japan.

Primary Examiner—James J. Seidleck

Assistant Examiner—U. K. Rajguru

Attorney, Agent, or Firm—Vivien Y. Tsang; Robert E. McDonald; Heidi A. Boehlefeld

ABSTRACT

[57] A coating composition has storage stability, and provides a highly durable coating on various substrates, such as wood. It can be a clear coating or a staining composition. The critical components of the composition are a self-curing vinyl polymer, an ammonia-neutralized polyurethane and a water-soluble catalyst to promote rapid curing under ambient conditions.

26 Claims, No Drawings

One Sheet

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DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L14</u>	polyisocyanate and tetra-n-butyl titanate and polyester and one component and composit\$5	10	<u>L14</u>
<u>L13</u>	polyisocyanate and tetra-n-butyl titanate and polyester and one component composit\$5	0	<u>L13</u>
<u>L12</u>	polyisocyanate and tetra-n-butyl titanate and polyester and composit\$5	53	<u>L12</u>
<u>L11</u>	polyisocyanate and tetra-n-butyl titanate and polyester and stoving composit\$5	0	<u>L11</u>
<u>L10</u>	polyisocyanate and tetra-n-butyl titanate and polyester	56	<u>L10</u>
<u>L9</u>	polyisocyanate and tetra-n-butyl titanate polyester	0	<u>L9</u>
<u>L8</u>	polyisocyanate and tetra-n-butyl titanate	61	<u>L8</u>
<u>L7</u>	polyisocyanate near15 malonic ester and tetra-n-butyl titanate	0	<u>L7</u>
<u>L6</u>	polyisocyanate near15 malonic ester	18	<u>L6</u>
<u>L5</u>	polyisocyanate near1 5 malonic ester	0	<u>L5</u>
<u>L4</u>	polyisocyanate near15 malonic ester	18	<u>L4</u>
<u>L3</u>	polyisocyanate near5 malonic ester	10	<u>L3</u>
<u>L2</u>	polyisocyanate near3 malonic ester	5	<u>L2</u>
<u>L1</u>	polyisocyanate near malonic ester	3	<u>L1</u>

END OF SEARCH HISTORY

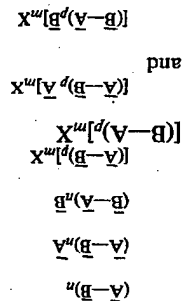
Typical examples of the dicarboxylic acids, and the derivatives thereof, used in the present invention are maleic acid, fumaric acid, chloromaleic acid, itaconic acid, cis-4-cyclohexene-1,2-dicarboxylic acid, endo-cis-bicyclo [2,2,1]-5-heptene-2,3-dicarboxylic acid and the anhydrides, the esters, the amides and the imides thereof. Preferable dicarboxylic acids, and the derivatives thereof, are maleic acid, fumaric acid and maleic anhydride. The most preferable one is maleic anhydride.

The modified block copolymers used in the present invention can be obtained by reacting the above-mentioned base block copolymers with the dicarboxylic acids, or the derivatives thereof, in a molten state or in a solution with or without using a free-radical initiator. Although there is no limitation in the production processes of the modified block copolymers which produce the modified block copolymers containing undesirable materials, such as gels or having a poor flowability, are not preferable for use in the present invention. For instance, as disclosed in the specification of prior co-pending application, U.S. Ser. No. 089,237, now U.S. Pat. No. 4,292,414, the modified block copolymers can be preferably produced in a manner such that the addition reaction can be conducted by using, for example, an extender in a molten state under the condition that no substantial amount of free radicals is generated in the system by using a free-radical inhibitor.

Further, the ionically crosslinked products of the modified block copolymers with at least one univalent, bivalent or trivalent metal ion can be used as the component A of the present thermoplastic polymer compositions. These ionically crosslinked modified block copolymers especially improve the impact resistance of the component B of the present thermoplastic polymer compositions. These ionically crosslinked modified block copolymers are obtained by crosslinking the modified block copolymers via ionic bondings with at least one metallic compound containing at least one univalent, bivalent or trivalent metal ion can be used as a crosslinking agent. In the ionically crosslinked modified block copolymers, the dicarboxylic acid groups, or the derivatives thereof, are ionized by the addition of the crosslinking agent compounds. The ionization degree or amount of the dicarboxylic acid groups, or the derivatives thereof, can be controlled by the addition amount of the crosslinking agent compounds. The ionization amount can be determined by the use of, for example, an infrared spectrophotometer.

The addition amount of the crosslinking agent compounds is determined so that the partial or the total amounts of the dicarboxylic acid groups, or the derivatives thereof, contained in the modified block copolymers are theoretically ionized. The ionization reaction proceeds substantially quantitatively. However, an excess amount of the crosslinking agents may be preferably used to obtain the desired ionization amount. In order to effectively obtain the ionically crosslinked modified block copolymers, the molar ratio of the metal components in the metallic compounds (i.e. the cross-

The block copolymers used in the present invention are generally prepared from the anionic copolymerization of the aromatic vinyl compounds and the conjugated diene compounds in an inert inactive hydrocarbon solvent, such as hexane, cyclohexane, benzene, toluene and the like, in the presence of, as a polymerization catalyst, an organic lithium compound, such as butyllithium. In the anionic copolymerization, the block copolymers having various structures can be obtained by changing the monomer addition method or order or by using a polyfunctional lithium compound. Furthermore, the micro structure of the conjugated diene portion of the block copolymer can be changed by the addition of a small amount of polar compounds, such as tetrahydropyran, diethylene glycol dimethyl ether and the like. In addition, the block copolymers having active lithium terminal groups, obtained from the above-mentioned methods, can be reacted with polyfunctional coupling agents, such as carbon tetrachloride, silicon tetrachloride and the like, to produce branched or radial type block copolymers. However, it should be noted that the block copolymers of the aromatic vinyl compounds and the conjugated diene compounds derived from any other production processes can be used in the present invention, so long as the molecular structure of the block copolymers are within the range of the above-mentioned limitations. Typical examples of the various structures of the block copolymers used in the present invention are as follows.



and

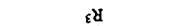
wherein A is a polymer block mainly containing the aromatic vinyl compound, B is a polymer block mainly containing the conjugated diene compound, X is a residual group of a polyfunctional coupling agent having two or more functional groups, n and p are, independently, integers of 1 or more and m is an integer of 2 or more. The modified block copolymers according to the present invention can be prepared by the addition reaction of unsaturated dicarboxylic acids, or the derivatives thereof, to the above-mentioned base block copolymers. The dicarboxylic acids, or the derivatives thereof, are addition reacted or grafted to the conjugated diene portions of the block copolymers at the active unsaturated positions thereof. These dicarboxylic acids, or the derivatives thereof, should be grafted to the block copolymer in an amount such that one or more dicarboxylic acid, or the derivatives thereof, on an average, are grafted to each molecule of the block copolymers and also such that 0.05 through 20 parts by weight, preferably 0.1 through 10 parts by weight, of the dicarboxylic acids, or the derivatives thereof, based on 100 parts by weight of the base block copolymer, are grafted to the block copolymer. When the amount of the grafted molecular units derived from the dicarbox-

<u>Set Name</u> side by side	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u> result set
<i>DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ</i>			
<u>L5</u>	tetra-n-butyl titanate and polyisocyanate and isobutanol	4	<u>L5</u>
<u>L4</u>	tetra-n-butyl titanate and polyisocyanate isobutanol	0	<u>L4</u>
<u>L3</u>	tetra-n-butyl orthotitanate and polyisocyanate	0	<u>L3</u>
<u>L2</u>	tetra-n-butyl titanate and polyisocyanate	61	<u>L2</u>
<u>L1</u>	tetra-n-butyl titanate	445	<u>L1</u>

END OF SEARCH HISTORY

one another, consisting of 2 to 6 carbon atoms. Also suitable

diglycidyl ether is also particularly suitable.



diglycidyl ether is also particularly suitable.

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Hit Count Set Name

result set

DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L7</u>	tetra near n near butyl titanate and polyisocyanate [clm]	17	<u>L7</u>
<u>L6</u>	tetra near n near butyl titanate and polyisocyanate [ab]	4	<u>L6</u>
<u>L5</u>	tetra near n near butyl titanate and polyisocyanate [ti]	0	<u>L5</u>
<u>L4</u>	tetra near n near butyl titanate and polyisocyanate	63	<u>L4</u>
<u>L3</u>	tetra near n near butyl titanate	537	<u>L3</u>
<u>L2</u>	tetra near n near butyl titanate and 6060573 [pn]	0	<u>L2</u>
<u>L1</u>	tetra-n-butyl titanate and 6060573 [pn]	0	<u>L1</u>

END OF SEARCH HISTORY

10/079661

20 Sep 2000
5 Sep 2000
①

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

Claims 4 and 9 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant

regards as the invention.

Claim 9 provides for the use of polyethylene as an antidrip agent in molding

compositions, but, since the claim does not set forth any steps involved in the method/process, it is unclear what method/process applicant is intending to encompass. A claim is indefinite where it merely recites a use without any active, positive steps delimiting how this use is actually

practiced.

Claim 9 is rejected under 35 U.S.C. 101 because the claimed recitation of a use, without

setting forth any steps involved in the process, results in an improper definition of a process, i.e., results in a claim which is not a proper process claim under 35 U.S.C. 101. See for example *Ex parte Dunki*, 153 USPQ 678 (Bd.App. 1967) and *Clinical Products, Ltd. v. Brenner*, 255 F.

Supp. 131, 149 USPQ 475 (D.D.C. 1966).

Claim 4 is indefinite since it is not known which type of av. Molecular weight is

envisioned (in lines 2-3).

WEST[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 4 of 4 returned.**☐ 1. Document ID: US H000752 H

L4: Entry 1 of 4

File: USPT

Mar 6, 1990

US-PAT-NO: H000752

DOCUMENT-IDENTIFIER: US H000752 H

TITLE: Magnetic recording medium

DATE-ISSUED: March 6, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Suzuki; Atsushi	Utsunomiya			JP
Koyama; Shigeru	Ichikai			JP

US-CL-CURRENT: 428/694BU; 428/694BS

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
Draw Desc	Image									

☐ 2. Document ID: US 4837245 A

L4: Entry 2 of 4

File: USPT

Jun 6, 1989

US-PAT-NO: 4837245

DOCUMENT-IDENTIFIER: US 4837245 A

TITLE: Process for the preparation of non-cellular or cellular polyurethane elastomers in the presence of a polyester polyol containing therein titanium and/or tin compounds as catalysts

DATE-ISSUED: June 6, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Streu; Joachim	Diepholz			DE
Straehle; Wolfgang	Heidelberg			DE
Ramlow; Gerhard	Weinheim			DE

US-CL-CURRENT: 521/117; 252/182.14, 252/182.3, 502/102, 502/151, 521/123, 521/127, 521/172, 521/173, 528/56, 528/58, 528/66

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
Draw Desc	Image									

Once the precipitate has been stabilized, residual water can be removed from the precipitate by centrifugation. The cellulose ester/functional additive blend is then dried by conventional techniques.

5 The invention further relates to a process for preparing a cellulose ester/functional additive blend, comprising:

(a) admixing the functional additive with the cellulose ester and a first acid;

(b) depositing the admixture of step (a) in a pellet;

(c) extruding the admixture from the pellet;

15 (d) immediately after step (c) or simultaneous with step (c), contacting the extruded admixture with a precipitating agent to precipitate the cellulose ester/functional additive to thereby produce an extrusion of

the cellulose ester/functional additive blend; and

20 (e) cutting the precipitated extrusion into pellets.

As described above, the admixture comprising the cellulose ester, the functional additive and the first acid are added to a bath containing a precipitating agent in order to coprecipitate the cellulose ester/functional additive blend. In

25 another embodiment of the invention, the admixture of step (a) can be added to a pellet prior to contacting the admixture with the precipitating agent. Pellets useful in the present invention are known in the art. In another embodiment, prior to adding the admixture of step (a) to the pellet, the admixture and pellet are heated. In one embodiment, the admixture is heated from 5 to 60°C, preferably, 5

30 to 15°C, and the pellet is heated from 5 to 60°C, preferably 5 to 15°C. In one

☐ 3. Document ID: US 4808477 A

L4: Entry 3 of 4

File: USPT

Feb 28, 1989

US-PAT-NO: 4808477

DOCUMENT-IDENTIFIER: US 4808477 A

TITLE: Urethane modified nylon magnet wire enamel

DATE-ISSUED: February 28, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Harber; Joseph J.	Fort Wayne	IN		

US-CL-CURRENT: 428/389; 427/117, 427/120, 427/388.1, 428/383, 428/458, 428/470

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 4. Document ID: US 4725458 A

L4: Entry 4 of 4

File: USPT

Feb 16, 1988

US-PAT-NO: 4725458

DOCUMENT-IDENTIFIER: US 4725458 A

TITLE: Urethane modified nylon magnet wire enamel

DATE-ISSUED: February 16, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Harber; Joseph J.	Fort Wayne	IN		

US-CL-CURRENT: 427/388.1; 427/120, 428/379, 428/383, 525/420, 525/424, 525/425

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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embodiment, prior to step (c) the temperature of the pellet is adjusted to

-5 to 25°C.

In one embodiment, the pellet containing the admixture of step (a) is

5 contacted with the precipitating agent. In another embodiment, the cutter end of the pellet is submerged into a bath comprising the precipitating agent. In one embodiment, the precipitating agent is water. In another embodiment, the precipitating agent comprises a second acid. Once the pellet extrudes the admixture of step (a) into the bath containing the precipitating agent, a pellet

10 comprising the cellulose ester/ functional additive is produced. In one embodiment, the precipitating agent is heated from 0 to 23°C prior to the contacting step.

Once the pellet of the cellulose ester/functional additive blend is produced, the pellets can be washed with water, treated with a stabilizer, and dried to remove residual water as described above for the powder process.

PREPARATION OF A CONTROLLED RELEASE MATRIX SYSTEM

20 In accordance with the purposes of this invention, as embodied and broadly described herein, the invention relates to a method for controlled release of an agricultural additive, comprising dispensing the controlled release matrix system, comprising:

25 (a) at least one biodegradable cellulose ester; and

(b) at least one agricultural additive,

wherein components (a) and (b) form a controlled release matrix system,

<u>Set Name</u> side by side	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u> result set
<i>DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ</i>			
<u>L5</u>	tetra-n-butyl titanate and polyisocyanate [clm]	17	<u>L5</u>
<u>L4</u>	tetra-n-butyl titanate and polyisocyanate [ab]	4	<u>L4</u>
<u>L3</u>	tetra-n-butyl titanate and polyisocyanate [ti]	0	<u>L3</u>
<u>L2</u>	tetra-n-butyl titanate and polyisocyanate	61	<u>L2</u>
<u>L1</u>	tetra-n-butyl titanate	445	<u>L1</u>

END OF SEARCH HISTORY

5,580,911; and 5,559,171 are incorporated herein by reference for a detailed discussion on biodegradability. Biodegradability can also occur by use of fungi.

5 Biodegradation of cellulose esters in a composting environment has been demonstrated. The factors that effect the rate of degradation are the type of

substituent (i.e. acetate, propionate or butyrate) and the degree of substitution of the cellulose. Typically, lower DS material degrades faster than high DS material and smaller substituents (acetate) degrade faster than larger ones.

10 A number of cellulose esters were synthesized from ^{14}C -labelled acetate and subjected to a composting environment. The release of $^{14}\text{CO}_2$ produced by degradation of the material was monitored and was used as an indicator of the degradation of the ester linkages. As illustrated by the figure below, the degradation of cellulose acetate with DS 1.85, as evidenced by the production of $^{14}\text{CO}_2$, is rapid with the majority of the material degrading within a week. Cellulose acetates with higher degrees of substitution require much longer periods of time to degrade. For example, cellulose acetate with DS 2.5 remains only partially degraded after 2 weeks time.

NAME	CITY	STATE	COUNTRY	RULE-47
Gindin, Lyubov K.	Pittsburgh	PA	US	
Henderson, Karen M.	Coraopolis	PA	US	
Lee, Sze-Ming	Houston	TX	US	
Long, Brian	Clarington	OH	US	

US-CL-CURRENT: 524/589; 524/839

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 5. Document ID: US 6444746 B1

L7: Entry 5 of 22

File: USPT

Sep 3, 2002

US-PAT-NO: 6444746

DOCUMENT-IDENTIFIER: US 6444746 B1

TITLE: Aqueous polyurethane dispersions containing pendant amide groups and their use in one-component thermoset compositions

DATE-ISSUED: September 3, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Gindin, Lyubov K.	Pittsburgh	PA		
Henderson, Karen M.	Coraopolis	PA		
Lee, Sze-Ming	Houston	TX		
Long, Brian	Clarington	OH		

US-CL-CURRENT: 524/591; 428/423.1, 524/839, 524/840

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 6. Document ID: US 6362300 B1

L7: Entry 6 of 22

File: USPT

Mar 26, 2002

US-PAT-NO: 6362300

DOCUMENT-IDENTIFIER: US 6362300 B1

TITLE: Moisture-curable polyurethane compositions

DATE-ISSUED: March 26, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Araki, Kiminori	Kanagawa			JP
Matsuda, Hideyuki	Kanagawa			JP
Nishi, Eiji	Kanagawa			JP
Samata, Takahiro	Kanagawa			JP

US-CL-CURRENT: 528/28; 524/115, 524/126, 524/425, 528/38, 528/58, 544/106

"Other polymer" or "another polymer" is defined as a polymer which is not included as one of the other functional additives listed herein. The polymers of this

25 After melt and/or solid phase polycondensation the polyesters have an inherent viscosity (I.V.) of about 0.65 to about 1.2 dL/g, preferably 0.75 dL/g measured at 25°C in a 60/40 ratio by weight of phenol/tetrachloroethane.

20 The term "functional additive" as used herein refers to cellulosic plastic modifiers. These modifiers can include, but are not limited to, plasticizers, other polymers, UV light stabilizers, dyes and pigments, acid stabilizers, agricultural chemicals, and bioactive compounds.

15 In this specification and in the claims which follow, reference will be made to a number of terms which shall be defined to have the following meanings:

As used in the specification and in the claims, "a" can mean one or more, depending upon the context in which it is used.

10 Before the present compositions of matter, products and methods are disclosed and described, it is to be understood that this invention is not limited to specific synthetic methods or to particular formulations, as such may, of course, vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting.

5 DETAILED DESCRIPTION OF THE INVENTION

general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed.

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 7. Document ID: US 6103849 A

L7: Entry 7 of 22

File: USPT

Aug 15, 2000

US-PAT-NO: 6103849

DOCUMENT-IDENTIFIER: US 6103849 A

TITLE: Storage stable, heat curable polyurethane compositions

DATE-ISSUED: August 15, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Squiller; Edward P.	Pittsburgh	PA		
Rosthauser; James W.	Glen Dale	WV		
Markusch; Peter H.	McMurray	PA		
Potter; Terry A.	Beaver	PA		

US-CL-CURRENT: 528/45; 528/59

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

☐ 8. Document ID: US 6063861 A

L7: Entry 8 of 22

File: USPT

May 16, 2000

US-PAT-NO: 6063861

DOCUMENT-IDENTIFIER: US 6063861 A

TITLE: Self crosslinkable polyurethane-polyacrylate hybrid dispersions

DATE-ISSUED: May 16, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Irle; Christoph	Krefeld			DE
Blum; Harald	Leverkusen			DE
Kremer; Wolfgang	Kerken			DE
Roschu; Rolf	Willich			DE

US-CL-CURRENT: 524/591; 428/423.1, 428/425.1, 524/507, 524/539, 524/839, 524/840, 525/123, 525/455

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

☐ 9. Document ID: US 6046270 A

invention are preferably polyesters. The polyesters of this invention can be any polyester known in the art, but is preferably an aliphatic polyester, or an aromatic-aliphatic copolyester. The polyesters of this invention may be miscible, partially miscible, or immiscible in certain combinations or compositions with the cellulose esters described herein. The polyesters of this invention may have an inherent viscosity greater than 0.40 dL/g and a preferred inherent viscosity of between 0.40 and 1.60 dL/g as measured at a concentration of 0.5 weight% in tetrachlorethane / phenol [40:60]. The polyesters may be prepared according to polyester forming conditions known in the art. The reaction should occur at a temperature to effect esterification and polycondensation. For example, a mixture of one or more aromatic or aliphatic dicarboxylic acids, preferably aliphatic dicarboxylic acids or ester forming derivatives thereof, and one or more diols may be heated in the presence of esterification and/or transesterification catalysts at temperatures in the range of about 150 C to 300 C, and more preferably in the range of about 200 C to 270 C. Normally, the dicarboxylic acid is esterified with the diol(s) at temperatures of 200 C to 270 C and elevated pressure under nitrogen. Polycondensation is then effected by increasing the temperature and lowering the pressure while excess diol(s) is removed from the mixture. The aliphatic polyesters of this invention may be prepared from diacids (or diesters) such as glutaric, adipic, succinic, and sebacic acids (or esters). Aliphatic-aromatic copolyesters may be prepared from the diacids (or diesters) above and aromatic diesters such as dimethyl terephthalate, dimethyl isophthalate and dimethyl 2,6-naphthalene dicarboxylate. These diacids and diesters may be polymerized with several diols such as ethylene glycol, butanediol, diethylene glycol, hexanediol and polyethylene glycol. Examples of polyesters suitable for this invention are: poly(ethylene glycol), poly(tetramethylene glutarate), poly(tetramethylene adipate), poly(hexamethylene glutarate), poly(dieethylene glutarate), poly(ethylene glutarate-co-terephthalate) [85/15], poly(ethylene glutarate-co-terephthalate) [70/30], poly(tetramethylene glutarate-co-

L7: Entry 9 of 22

File: USPT

Apr 4, 2000

US-PAT-NO: 6046270
DOCUMENT-IDENTIFIER: US 6046270 A

TITLE: Silane-modified polyurethane resins, a process for their preparation and their use as moisture-curable resins

DATE-ISSUED: April 4, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Roesler; Richard R.	Wexford	PA		
Schmalstieg; Lutz	Cologne			DE

US-CL-CURRENT: 524/590; 524/588, 524/589, 528/28, 528/44, 528/60, 528/85

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 10. Document ID: US 5710209 A

L7: Entry 10 of 22

File: USPT

Jan 20, 1998

US-PAT-NO: 5710209
DOCUMENT-IDENTIFIER: US 5710209 A

TITLE: Aqueous binders for coating compositions based on polyester polyurethanes

DATE-ISSUED: January 20, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Blum; Harald	Wachtendonk			DE
Pedain; Josef	Koln			DE

US-CL-CURRENT: 524/591; 524/538, 524/839, 524/840

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 11. Document ID: US 5656712 A

L7: Entry 11 of 22

File: USPT

Aug 12, 1997

US-PAT-NO: 5656712
DOCUMENT-IDENTIFIER: US 5656712 A

TITLE: Polyurethane compositions and method

DATE-ISSUED: August 12, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mirossay; Thomas C.	Arlington	TX	76018	

terephthalate) [85/15], poly(tetramethylene adipate-co-terephthalate) [70/30], poly(tetramethylene glutarate-co-terephthalate) [70/30], poly(tetramethylene-co-ethylene glutarate-co-terephthalate) [50/50;85/15], poly(tetramethylene glutarate-co-terephthalate) [50/50; 70/30], poly(hexamethylene glutarate-co-terephthalate) [75/25], poly(hexamethylene glutarate-co-terephthalate) [70/30], poly(ethylene-co-polyethylene glutarate) [91/9], poly(ethylene-co-polyethylene glutarate-co-terephthalate) [91/9;70/30]. The polyesters of this invention may be those prepared biologically, such as polyhydroxybutyrate or copolymers of polyhydroxybutyrate and polyhydroxyvalerate.

The polyesters may be prepared according to polyester forming conditions well known in the art. The reaction should occur at a temperature to effect esterification and polycondensation. For example, a mixture of one or more dicarboxylic acids, preferably aromatic dicarboxylic acids, or ester forming derivatives thereof, and one or more diols may be heated in the presence of esterification and/or transesterification catalysts at temperatures in the range of about 150° to about 300°C, preferably, about 200°C to about 300°C, and even more preferably, about 260°C to about 300°C, and pressures of atmospheric to about 0.2 mm Hg. Normally, the dicarboxylic acid is esterified with the diol(s) at elevated pressure and at a temperature at about 240°C to about 270°C. Polycondensation then is effected by increasing the temperature and lowering the pressure while excess diol is removed from the mixture.

25 The term "degree of substitution" as used herein refers to the number of substituents per anhydroglucose unit where the maximum DS/AGU is three. The term "degree of substitution" will also be referred to as "DS" or DS/AGU" throughout the application.

US-CL-CURRENT: 528/56; 528/61, 528/905

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	RWC
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☐ 12. Document ID: US 5610232 A

L7: Entry 12 of 22

File: USPT

Mar 11, 1997

US-PAT-NO: 5610232

DOCUMENT-IDENTIFIER: US 5610232 A

TITLE: Aqueous non-gelling, anionic polyurethane dispersions and process for their manufacture

DATE-ISSUED: March 11, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Duan; Youlu	Minneapolis	MN		
Stammler; Sonja	St. Croix	MN		
Rhein; Scott	Forest Lake	MN		
Lindquist; Lowell	St. Paul	MN		

US-CL-CURRENT: 524/840; 156/331.1, 156/331.4, 156/331.7, 428/423.1, 428/423.4, 428/423.7, 428/424.2, 428/424.6, 428/424.8, 428/425.1, 428/425.8, 524/500, 524/507, 524/591, 525/123, 525/127, 525/440, 525/454, 525/455, 525/457, 525/528, 528/71, 528/905

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	RWC
Draw Desc	Image									

☐ 13. Document ID: US 5608000 A

L7: Entry 13 of 22

File: USPT

Mar 4, 1997

US-PAT-NO: 5608000

DOCUMENT-IDENTIFIER: US 5608000 A

TITLE: Aqueous polyurethane dispersion adhesive compositions with improved heat resistance

DATE-ISSUED: March 4, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Duan; Youlu	Minneapolis	MN		
Stammler; Sonja	Marine On the St. Croix	MN		

US-CL-CURRENT: 524/591; 428/423.1, 428/423.4, 428/423.7, 428/424.2, 428/424.6, 428/424.8, 428/425.1, 428/425.8, 524/500, 524/507, 524/840, 525/123, 525/127, 525/440, 525/453, 525/454, 525/455, 525/457, 525/528, 528/71, 528/905

PREPARATION OF A CELLULOSE ESTER/FUNCTIONAL ADDITIVE
BLEND BY COPRECIPITATION

5 In accordance with the purpose(s) of this invention, as embodied and broadly described herein, the invention, in one aspect, relates to a process for blending a cellulose ester with a functional additive, comprising:

(a) admixing the functional additive with the cellulose ester and a first acid, and

(b) contacting the admixture with an aqueous precipitating agent, whereby a blend comprising the cellulose ester and the functional additive coprecipitates.

15 The invention further relates to a process for blending a cellulose ester with a functional additive, comprising:

(a) admixing

i) a functional additive comprising a plasticizer, another polymer, a UV light stabilizer, a dye, a pigment, an acid stabilizer, a flame retardant, an agricultural chemical, bioactive compound or a mixture thereof,

25 ii) a cellulose ester comprising cellulose acetate, cellulose acetate phthalate, cellulose acetate butyrate, cellulose butyrate, cellulose propionate, cellulose acetate propionate, acetate, carboxymethylcellulose acetate propionate,

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
Draw Desc	Image									

☐ 14. Document ID: US 5548057 A

L7: Entry 14 of 22

File: USPT

Aug 20, 1996

US-PAT-NO: 5548057

DOCUMENT-IDENTIFIER: US 5548057 A

TITLE: One-component-type composition for polyurethane elastomer and method for processing the same by molding

DATE-ISSUED: August 20, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Hirayama; Shinji	Hadano			JP
Konishi; Shin	Fujisawa			JP
Hidai; Takao	Anjo			JP
Hama; Shinjiro	Fujisawa			JP
Hashimoto; Sadako	Hadano			JP
Morikawa; Yukihiro	Yokohama			JP

US-CL-CURRENT: 528/67

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
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☐ 15. Document ID: US 5142014 A

L7: Entry 15 of 22

File: USPT

Aug 25, 1992

US-PAT-NO: 5142014

DOCUMENT-IDENTIFIER: US 5142014 A

TITLE: Ambient temperature curable one-component polyurethane or polyurea compositions

DATE-ISSUED: August 25, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Markusch; Peter H.	McMurray	PA		
Squiller; Edward P.	Wheeling	WV		

US-CL-CURRENT: 528/45; 528/44, 528/60, 528/62, 528/64, 528/71

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
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☐ 16. Document ID: US 5138011 A

L7: Entry 16 of 22

File: USPT

Aug 11, 1992

carboxymethylcellulose acetate butyrate, cellulose acetate butyrate succinate, or a mixture thereof, and

- 5 (iii) a first acid comprising acetic acid, propionic acid, butyric acid or a mixture thereof, and

- (b) contacting the admixture with an aqueous precipitating agent comprising water, acetic acid, propionic acid, butyric acid, or a mixture thereof, whereby a blend comprising the cellulose ester and the functional additive coprecipitates.
- 10

The invention relates to a method of producing a blend of a cellulose ester and a functional additive by coprecipitation from carboxylic acid(s) dopes, into water or aqueous carboxylic acid(s). As used herein, the term "coprecipitation" refers to the act of causing two or more chemicals or chemical compounds in an admixture solution or suspension in the presence of a solvent or solvent mixture to precipitate by the addition of a precipitant, such that the greater fraction of the desired chemicals or chemical compounds are incorporated into the resulting precipitate phase.

20

In one embodiment, a functional additive can be incorporated into the precipitated cellulose ester blend. In a particular embodiment, the functional additive can be a plasticizer, another polymer, a UV light stabilizer such as organic phosphites known in the art, a dye or a pigment, an acid stabilizer, a flame retardant, an agricultural chemical (i.e. pesticide, herbicide, fertilizer, trace mineral), a bioactive compound (i.e. medicaments), or a mixture thereof. In a more preferred embodiment, the functional additive is a plasticizer, a UV stabilizer, a dye, or a mixture thereof.

25

US-PAT-NO: 5138011

DOCUMENT-IDENTIFIER: US 5138011 A

TITLE: One-component polyurethane or polyurea compositions

DATE-ISSUED: August 11, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Markusch; Peter H.	McMurray	PA		
Squiller; Edward P.	Wheeling	WV		

US-CL-CURRENT: 528/45; 528/44, 528/52, 528/64

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 17. Document ID: US 5124447 A

L7: Entry 17 of 22

File: USPT

Jun 23, 1992

US-PAT-NO: 5124447

DOCUMENT-IDENTIFIER: US 5124447 A

TITLE: Ambient temperature curable one-component polyurethane or polyurea compositions

DATE-ISSUED: June 23, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Squiller; Edward P.	Wheeling	WV		
Markusch; Peter H.	McMurray	PA		

US-CL-CURRENT: 528/45; 523/414, 523/415, 523/417, 528/60, 528/68, 528/73

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 18. Document ID: US 4425468 A

L7: Entry 18 of 22

File: USPT

Jan 10, 1984

US-PAT-NO: 4425468

DOCUMENT-IDENTIFIER: US 4425468 A

TITLE: Polyurea-polyurethane acrylate polymer dispersions

DATE-ISSUED: January 10, 1984

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Makhlouf; Joseph M.	Mars	PA		
McCollum; Gregory J.	Glenshaw	PA		
Kerr; Paul R.	Allison Park	PA		

Examples of plasticizers suitable for the present invention include, but are not limited to, dioctyl adipate, triethylene glycol-2-ethylhexanoate, polyethylene glutarate, dioctyl phthalate, diethyl phthalate, butyl benzyl phthalate, triethyl citrate, tripropionin, polypropylene glycol dibenzoate, polyethylene succinate, sucrose acetate isobutyrate, triphenyl phosphate, polyalkyl glycoside, triethyl phosphate, diethyl phthalate, 2,2,4-trimethyl-1,3-pentane-diol diisobutyrate, a copolymer of phthalic acid, 1,3-butanediol, and 1,4-butanediol end capped by aliphatic epoxide, or a mixture thereof.

10 Examples of UV stabilizers and antioxidants suitable for the present invention include, but are not limited to, epoxides of a natural oil, and mineral oil, organic phosphites, or a mixture thereof.

15 Examples of organic dyes suitable for the present invention include, but are not limited to, C.I. Solvent Violet 13, C.I. Pigment Blue 15, C.I. Pigment Blue 28, C.I. Dispersion Violet 8, and C.I. Pigment Red 122. A preferred dye is C.I. Solvent Violet 13.

20 A wide variety of agricultural additives can be used in the present invention. In one embodiment, the agricultural additive comprises an insecticide, a herbicide, a pesticide, a fertilizer, a trace mineral, or a mixture thereof. In another embodiment, the agricultural additive is an insecticide comprising an organochlorine compound, an organophosphate compound, an aryl compound, a heterocyclic compound, an organosulfur compound, a carbamate compound, a formamidine compound, a dinitrophenol compound, an organotin compound, a pyrethroid compound, an acylurea compound, a botanical compound, an antibiotic compound, a fumigant compound, a repellent compound, an inorganic compound, or a mixture thereof.

US-CL-CURRENT: 524/710; 524/840, 525/440, 525/455, 525/459, 525/920, 526/75, 528/75

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 19. Document ID: US 4373081 A

L7: Entry 19 of 22

File: USPT

Feb 8, 1983

US-PAT-NO: 4373081

DOCUMENT-IDENTIFIER: US 4373081 A

TITLE: Coating compositions and process for the production of polyurethane coatings

DATE-ISSUED: February 8, 1983

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Nachtkamp; Klaus	Cologne			DE
Bock; Manfred	Leverkusen			DE
Mennicken; Gerhard	Leverkusen			DE
Pedain; Josef	Cologne			DE

US-CL-CURRENT: 528/45; 428/423.1, 428/425.8

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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☐ 20. Document ID: US 4173691 A

L7: Entry 20 of 22

File: USPT

Nov 6, 1979

US-PAT-NO: 4173691

DOCUMENT-IDENTIFIER: US 4173691 A

TITLE: Catalyst composition for flexible polyurethane foams

DATE-ISSUED: November 6, 1979

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Treadwell; Kenneth	Rahway	NJ		

US-CL-CURRENT: 521/124; 502/170, 521/125

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
Draw Desc	Image									

☐ 21. Document ID: US 3953169 A

L7: Entry 21 of 22

File: USPT

Apr 27, 1976

- In another embodiment, the organochlorine compound comprises a diphenyl aliphatic compound; hexachlorocyclohexane; a cycloiene; or a polychloroterpene. In one embodiment, the diphenyl aliphatic compound comprises 1,1-dichloro-2,2-bis(p-chlorophenyl)ethane; 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane; dicofol; ethylan; chlorbenzilate; or methoxychlor. In another embodiment, the cycloiene comprises chlordane; aldrin; dieldrin; heptachlor; endrin; mirex; endosulfan; or chlordane. In another embodiment, the polychloroterpene comprises toxaphene or strobane. In another embodiment, the organophosphate comprises an aliphatic phosphate compound; an aryl phosphate compound; or a heterocyclic phosphate compound. Examples of aliphatic compounds include, but are not limited to, malathion; trichlorofon; monocrotophos; dimethoate; oxydemetonmethyl; dicrotophos; disulfoton; dichlorvos; mevinphos; methamidophos; or acephate. Examples of phenyl compounds include, but are not limited to, ethyl parathion; methyl parathion; profenofos; sulprofos; isofenphos; fenitrothion; fenithion; or famphur. Examples of heterocyclic compounds include, but are not limited to, diazinon; azinphos-methyl; chlorpyrifos; methidathion; phosmet; isazophos; chlorpyrifos-methyl; or azinphos-ethyl. In another embodiment, the organosulfur compound comprises tetradifon; propargite or over. In another embodiment, the carbamate comprises carbaryl; methomyl; carbosulfan; aldicarb; oxamyl; thiodicarb; methiocarb; propoxur; bendiocarb; carbosulfan; aldoxycarb; trimethacarb; promecarb; or fenoxycarb. In another embodiment, the formamide comprises chlordimeform; formetanate; or amitraz. In another embodiment, the dinitrophenol compound comprises binapacryl or dinocap. In another embodiment, the organotin compound comprises cyhexatin or fenbutatin-oxide. In another embodiment, the pyrethroid comprises allethrin; tetramethrin; bioresmethrin; bioallethrin; phonothrin; fenvalerate; permethrin; bifenthrin; lambda cyhalothrin; cypermethrin; cyfluthrin; delta methrin esfenvalerate; fenpropathrin; flucythrinate; fluralinate; prallethrin; or talomethrin. In another embodiment, the acylurea comprises triflumuron; chlorfluazuron; teflubenzuron; hexaflumuron; flufenoxuron;

US-PAT-NO: 3953169

DOCUMENT-IDENTIFIER: US 3953169 A

TITLE: Paperboard impregnated with polyurethane resin

DATE-ISSUED: April 27, 1976

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Igarashi; Seiichi	Suita			JA
Shirakawa; Kin-ichi	Ibaraki			JA
Kimura; Kazuo	Kobe			JA
Ogino; Akira	Shinsenri-Higashi			JA

US-CL-CURRENT: 8/192; 428/425.1, 428/537.5, 528/73, 528/77, 528/83, 8/DIG.11

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMIC
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☐ 22. Document ID: US 3886226 A

L7: Entry 22 of 22

File: USPT

May 27, 1975

US-PAT-NO: 3886226

DOCUMENT-IDENTIFIER: US 3886226 A

TITLE: Polyurethane composition

DATE-ISSUED: May 27, 1975

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Asai; Kiyotsugu	Kawasaki			JA
Kawabata; Toshihiko	Tokyo			JA
Sakai; Koichi	Tokyo			JA
Fukuda; Kiyo	Yokohama			JA
Nagahisa; Seiji	Kawasaki			JA
Ichikawa; Toshiyuki	Tokyo			JA

US-CL-CURRENT: 528/28; 428/425.6, 428/429, 525/440, 525/446, 528/26, 528/29, 528/38,
528/49, 528/61, 528/65, 528/68

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMIC
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- flucycloxuron; or novaluron. In another embodiment, the botanical compound comprises pyrethrum; nicotine; camphor; turpentine; rotenone; limonene; or neem oil. In another embodiment, the antibiotic comprises avermectins. In another embodiment, the fumigant comprises methyl bromide; ethylene dichloride; sulfur fluoride; chloroethene; naphthalene; or paradichlorobenzene. In another embodiment, the repellent comprises dimethyl phthalate; dibutyl phthalate; benzyl benzoate; N-butyl acetanilide; dimethyl carbate; or diethyl toluamide. In another embodiment, the inorganic compound comprises sulfur; mercury; thallium; antimony; copper arsenate; inorganic fluorides; boric acid; disodium octaborate; or silica gels.
- 10 In another embodiment, the agricultural additive is a herbicide comprising an ALSase inhibitor, an aromatic carboxylic acid, chloroacetamide, a triazine, an ESPSase inhibitor, an ACCase inhibitor, dinitroaniline compound, bentazon, a haloxydroxybenzonitrile, a diphenyl ether, an isoxazolidone, paraquat or a mixture thereof.
- 15 In another embodiment, the ALSase inhibitor comprises a sulfonamide, a sulfonamide, or a triazopyrimidine sulfonamide. Examples of sulfonamides include, but are not limited to, chlorosulfuron; chlorsulfuron-ethyl; nicosulfuron; primisulfuron; thifensulfuron; metsulfuron; sulfometuron-methyl; or bensulfuron-methyl. Examples of imidazolinones include, but are not limited to, imazethapyr; imazethapyr; or imazamethabenz. An example of a triazopyrimidine sulfonamide includes, but is not limited to, flumetsulam. In another embodiment, the aromatic carboxylic acid comprises a phenoxycetic acid, a benzoic acid, or an aryloxyphenoxycetic acid. Examples of phenoxycetic acids include, but are not limited to, 2,4-dichlorophenoxycetic acid (2,4-D); or 2,4,5-trichlorophenoxycetic acid (2,4,5-T). Examples of benzoic acids include, but are not limited to, chloramben. Examples of aryloxyphenoxycetic acids include, but are not limited to, chloramben.
- 20 25

Term	Documents
ONE.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	7211299
ONES.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	343150
COMPONENT.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1766486
COMPONENTS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1938757
COMPOSITION.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1408429
COMPSN.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	449324
COMPSNS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	113691
COMPOSITIONS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	469379
POLYURETHANE[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	50716
POLYURETHANES[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	2475
POLYISOCYANATE.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	52877
(ONE COMPONENT COMPOSITION AND POLYURETHANE [TI] AND POLYISOCYANATE AND POLYESTER).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	22

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are not limited to, diclofop-methyl, fluazifop-butyl, or quizalafop-ethyl. In another embodiment, the chloroacetamide comprises alachlor, metolachlor, propachlor, butachlor, diphenamide, napropamide, pronamide, propanil, or acetochlor. In another embodiment, the triazine comprises a chlorinated s-triazine; a methoxy s-triazine; a methylthio s-triazine; or an asymmetrical triazine. Examples of

chlorinated s-triazines include, but are not limited to, atrazine, cyanazine, cyprozin, simazine, procyzine, or propazine. Examples of methoxy s-triazines include, but are not limited to, atron, prometon, secbumeton, or simeton. Examples of methylthio s-triazines include, but are not limited to, ametryn;

prometryn; terbuthryn; simetryn; or desmetryn. An example of an asymmetrical triazine includes, but is not limited to, Metribuzin. An example of an ESPSase inhibitor includes, but is not limited to, glyphosphate. In another embodiment, the

ACCase inhibitor comprises an aryloxyphenoxypionate or a cyclohexenone. Examples of aryloxyphenoxypionates include, but are not limited to, diclofop-

methyl, fluazifop-butyl, or quizalafop-ethyl. Examples of cyclohexenones include, but are not limited to, sethoxydim, clethodim, allyoxydim, or cycloxydim. In

another embodiment, the dinitroaniline compound comprises a methylaniline

herbicide or a sulfonylaniline. Examples of methylaniline herbicides include, but

are not limited to, trifluralin, pendimethalin, benefin, dinitramine, fluchloralin, or

profluralin. Examples of sulfonylaniline compounds include, but are not limited to,

oryzalin or nitralin. In another embodiment, the haloxydroxybenzonitrile

comprises bromoxynil or ioxynil. In another embodiment, the isoxazolidone

comprises clomazone.

The advantages of the present invention vary with the particular application.

In cellulosic plastics manufacturing, the application of this invention provides the economic benefit of fewer processing steps in addition to fewer heat histories in the production of the cellulosic plastic materials. Moreover, the process of the present

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COMPONENT.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1766486
COMPONENTS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1938757
COMPOSITION.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1408429
COMPSN.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	449324
COMPSNS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	113691
COMPOSITIONS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	469379
POLYURETHANE[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	50716
POLYURETHANES[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	2475
POLYISOCYANATE.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	52877
(ONE COMPONENT COMPOSITION AND POLYURETHANE [TI] AND POLYISOCYANATE AND POLYESTER).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	22

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5 Another advantage of the present invention with respect to agrochemicals involves the production of a granular material with timed and sustained release properties, lower handling toxicity by virtue of reduced dusting and encapsulation of the functional additive, and increased UV light stability or hydrolytic stability of sensitive materials.

The process of the invention comprises adding a suitable functional additive,
or additives package, to a solution of a first carboxylic acid and a cellulose ester. In
one embodiment, the cellulose ester can be cellulose acetate, cellulose acetate
phthalate, cellulose acetate butyrate, cellulose propionate,
cellulose acetate propionate, cellulose propionate butyrate, carboxymethylcellulose
acetate, carboxymethylcellulose acetate propionate, carboxymethylcellulose acetate
butyrate, cellulose acetate succinate or a mixture thereof. In a preferred
embodiment, the cellulose ester is cellulose acetate, cellulose acetate propionate, or
a mixture thereof. In one embodiment, the degree of substitution of the cellulose
acetate is from 0.5 to 3.0, preferably 1.5 to 2.8, more preferably 1.8 to 2.2. In
another embodiment, the cellulose acetate propionate has a degree of substitution of
propionyl of from 0.1 to 3.0, preferably from 1.5 to 2.0. The degree of substitution
of acetyl is from 0.01 to 1.0, preferably 0.05 to 0.5.

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<u>L7</u>	one component composition and polyurethane [ti] and polyisocyanate and polyester	22	<u>L7</u>
<u>L6</u>	one component composition and polyurethane [ti] and polyisocyanate	29	<u>L6</u>
<u>L5</u>	one component composition and polyurethane [ti] and plyisocyanate	0	<u>L5</u>
<u>L4</u>	one component composition and polyurethane [ti] and tetra-n-butyl titanate	0	<u>L4</u>
<u>L3</u>	one component composition and polyurethane [ti]	37	<u>L3</u>
<u>L2</u>	one component composition and polyurethane	222	<u>L2</u>
<u>L1</u>	one component composition	932	<u>L1</u>

END OF SEARCH HISTORY

The selection of the first acid can vary depending upon the end-use of the

resulting cellulose ester blend. The mixture of carboxylic acid and water is chosen to dissolve the cellulose ester and functional additives. In one embodiment, the

5 first acid is a carboxylic acid. In another embodiment, the first acid is an aqueous

carboxylic acid. In a preferred embodiment, the first carboxylic acid is acetic acid,

propionic acid, butyric acid, or a mixture thereof, optionally, containing an amount

of water in sufficient quantities to dissolve the cellulose ester and functional

additives. Preferably, the first acid is present in the amount of 60 to 90 % by

10 weight and the water is from 2 to 15 % by weight of the admixture in step (a).

More preferably, the first acid is present in the amount of 10 to 90 % by

weight propionic acid or butyric acid to 30 % by weight water.

15 The functional additive is present in the amount of 1 to 50 % by weight,

preferably 1 to 20 % by weight, of the cellulose ester of step (a).

In one embodiment, the functional additive is added to a solution of the

cellulose ester and first carboxylic acid followed by stirring the admixture to

20 dissolve the functional additive to make a homogeneous solution. As described

above, preferred functional additives include plasticizers, UV stabilizers, and dyes.

In one embodiment, the amount of the plasticizer(s) is from 1 to 40 % by weight of

the cellulose ester in step (a), preferably from 15 to 25 % by weight of the

plasticizer. In a preferred embodiment, the functional additive is a plasticizer.

Once the cellulose ester and functional additive have been dissolved, the

admixure is contacted with an aqueous precipitating agent in order to coprecipitate

the cellulose ester/functional additive blend. The term "aqueous precipitating

agent" is defined as a solution comprising water and, optionally, one or more other

30 components. In one embodiment, the aqueous precipitating agent is water. In

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L7: Entry 1 of 22

File: PGPB

Aug 1, 2002

PGPUB-DOCUMENT-NUMBER: 20020103292

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020103292 A1

TITLE: Aqueous polyurethane dispersions containing modified hydrazide groups and their use in one-component thermoset compositions

PUBLICATION-DATE: August 1, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Blum, Harald	Leverkusen	PA	DE	
Gindin, Lyubov K.	Pittsburgh	PA	US	
Henderson, Karen M.	Coraopolis	PA	US	
Jacobs, Patricia B.	Pittsburgh	TX	US	
Lee, Sze-Ming	Houston	OH	US	
Long, Brian	Clarrington	PA	US	
Sylvester, Robert A.	Carnegie	PA	US	
Wicks, Douglas A.	Mt. Lebanon		US	

US-CL-CURRENT: 524/845, 523/400, 523/402, 523/414, 523/415, 524/501, 524/502, 524/507, 524/555, 524/588, 524/589, 524/590, 524/591, 524/593, 524/596, 524/600, 524/606, 524/608, 524/612, 524/816, 524/831, 524/838, 524/839, 524/840, 524/858, 524/860, 524/864, 524/869, 524/871, 524/877, 524/879, 525/123, 525/124, 525/329.4, 525/418, 525/474, 525/504, 525/509, 525/523, 525/528

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
Draw Desc	Image										

☐ 2. Document ID: US 20020068800 A1

L7: Entry 2 of 22

File: PGPB

Jun 6, 2002

PGPUB-DOCUMENT-NUMBER: 20020068800

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20020068800 A1

TITLE: Polyurethanes containing secondary amide groups and their use in one-component thermoset compositions

PUBLICATION-DATE: June 6, 2002

INVENTOR-INFORMATION:

wherein the blend is a controlled release matrix system.

5 Additionally, this invention relates to a method for controlled release of an agricultural additive comprising dispensing the controlled release matrix system, further comprising:

(a) at least one biodegradable cellulose ester; and

10 (b) at least one agricultural additive,

wherein components (a) and (b) form a controlled release matrix system,

in the proximity of the target for the additive and for a period of time

15 sufficient to undergo biodegradation and release the additive.

This invention also relates to a method for controlled release of a

pharmaceutical additive in the proximity of a target for the additive, comprising dispensing the controlled release matrix system, comprising:

20 (a) at least one biodegradable cellulose ester; and

(b) at least one pharmaceutical additive,

25 wherein components (a) and (b) form a controlled release matrix system,

in the proximity of the target for the additive and for a period of time sufficient to undergo biodegradation and release the additive.

NAME	CITY	STATE	COUNTRY	RULE-47
Gambino, Charles A.	McDonald	PA	US	
Henderson, Karen M.	Coraopolis	PA	US	
Jacobs, Patricia B.	Pittsburgh	PA	US	
Lee, Sze-Ming	Houston	TX	US	
Sylvester, Robert A.	Carnegie	PA	US	

US-CL-CURRENT: 525/456

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
Draw Desc	Image										

☐ 3. Document ID: US 20020068790 A1

L7: Entry 3 of 22

File: PGPB

Jun 6, 2002

PGPUB-DOCUMENT-NUMBER: 20020068790
PGPUB-FILING-TYPE: new
DOCUMENT-IDENTIFIER: US 20020068790 A1

TITLE: Aqueous polyurethane dispersions containing pendant amide groups and their use
in one-component thermoset compositions

PUBLICATION-DATE: June 6, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Gindin, Lyubov K.	Pittsburgh	PA	US	
Henderson, Karen M.	Coraopolis	PA	US	
Lee, Sze-Ming	Houston	TX	US	
Long, Brian	Clarington	OH	US	

US-CL-CURRENT: 524/589; 524/839

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMC
Draw Desc	Image									

☐ 4. Document ID: US 20020068789 A1

L7: Entry 4 of 22

File: PGPB

Jun 6, 2002

PGPUB-DOCUMENT-NUMBER: 20020068789
PGPUB-FILING-TYPE: new
DOCUMENT-IDENTIFIER: US 20020068789 A1

TITLE: Aqueous polyurethane dispersions containing secondary amide groups and their use
in one-component thermoset compositions

PUBLICATION-DATE: June 6, 2002

INVENTOR-INFORMATION:

In addition, this invention further relates to a controlled release matrix system, comprising a homogeneous mixture of:

(a) at least one biodegradable cellulose ester; and

(b) at least one agricultural additive or pharmaceutical additive,

wherein components (a) and (b) form a controlled release matrix system.

This invention further relates to a controlled release matrix system, consisting essentially of a homogeneous mixture of:

(a) at least one biodegradable cellulose ester; and

(b) at least one agricultural additive or pharmaceutical additive,

wherein components (a) and (b) form a controlled release matrix system.

20 This invention provides for an efficient method of preparing cellulose ester blends that contain functional additives. It also provides a controlled release matrix system than can release a functional additive.

25 Additional advantages of the invention will be set forth in part in the description which follows, and in part will be obvious from the description, or may be learned by practice of the invention. The advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims. It is to be understood that both the foregoing